

GEORGIA INSTITUTE OF TECHNOLOGY

ENGINEERING EXPERIMENT STATION

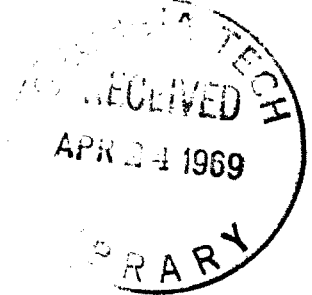
ATLANTA, GEORGIA 30332

January 14, 1965

State Highway Department of Georgia
2 Capitol Square, S. W.
Atlanta, Georgia

Attention: Mr. H. H. Huckeba
State Highway Planning Engineer

Subject: Monthly Progress Report No. 1
Georgia Tech Project No. A-802
"Hot Melt Traffic Marking Materials."
Covering the period 1 December 1964 to
31 December 1964



Gentlemen:

Project activities in December have included:

1. State of the Art

Copies of various reports, patents, publications, and specifications have been accumulated, and are being studied on a continuing basis. Of particular interest was a "Summary of Experimental Installations of Thermoplastic Pavement Striping Materials," dated April 13, 1964, supplied by the Bureau of Public Roads Office of Research and Development. Since this survey provided much information regarding current practices and experiences of various highway authorities, the plan to circulate a questionnaire seeking similar information has been deferred.

Project personnel have visited with officials of the Traffic Department of the City of Atlanta to discuss hot melt markings, and have also examined the equipment and application procedures in close detail.

2. Equipment and Procedures Development

On the basis of the examinations of full scale equipment and procedures, a simple lab scale applicator was designed and constructed. This device proved capable of applying uniform hot melt stripes to test discs for evaluation work on the accelerated wear tester in the laboratory.

Commercial equipment for full scale hot melt application does not lend itself well to field application of short lengths of a number of different hot melt formulations as will be required for project field studies. For this purpose, an applicator is being designed to permit rapid changeover with small quantities of hot melt composition.

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In addition to the applicators, suitable simple equipment for mixing and melting of materials is being adopted for project use.

3. Formulation Studies

Field experience to date has involved, predominantly, proprietary materials. Accordingly, it appeared worthwhile to undertake some characterization of these items as representative of "workable" compositions. Viscosity versus temperature curves have been determined for specimens of Permaline and Catatherm hot melts. Distinct differences in the materials were quite evident. At 400° F Permaline exhibited a consistency of 12,000 cps., whereas Catatherm was significantly higher at about 18,000 cps. Further work has been undertaken on these materials to determine approximate levels of resin content, beading and pigmentation. Laboratory apparatus is being assembled for determination of other physical properties as outlined in the proposal.

4. Short Range Plans

Special emphasis is being directed toward achieving initial field applications as soon as possible. Accordingly, project effort is presently being concentrated on construction of application equipment, and on preparing several experimental hot melt compositions in a relatively uncritical fashion. More critical laboratory formulations and evaluation work will be undertaken during the period that initial field tests are maturing.

Respectfully submitted:

W. R. Tooke, ~~Jr.~~
Project Director

Approved:

W. H. Burrows, Head
Industrial Products Branch

GEORGIA INSTITUTE OF TECHNOLOGY

ENGINEERING EXPERIMENT STATION

ATLANTA, GEORGIA 30332

February 8, 1965

Division of Highway Planning
State Highway Department of Georgia
Atlanta, Georgia

Attention: Mr. H. H. Huckeba
State Highway Planning Engineer

Subject: Monthly Progress Letter No. 2, Project A-802
"Hot Melt Traffic Marking Materials"
Contract No. 6401
Covering the period from January 1 to January 31, 1965



Gentlemen:

Project activities in January have included:

1. State of the Art. A formal patent search has been initiated. Study of the published literature is continuing, and project personnel have conferred with various resin suppliers concerning materials of interest.

2. Equipment and Procedures Development. Laboratory testing apparatus is being accumulated, and design of field application equipment is nearing completion.

3. Formulation Studies. In addition to hot melt viscosity characterization, proprietary materials have been examined to determine resin, beading, and pigmentation content. The California hot melt specification has been prepared and the melt viscosity of the resin binder and the complete formulation have been characterized. This material appears to work very satisfactorily with laboratory application procedures.

4. Immediate Plans. Priority of effort continues to be directed toward initiating highway test applications at the earliest possible date.

Respectfully submitted:

W. R. Tooke, Jr.
Project Director

Approved:

W. H. Burrows, Head
INDUSTRIAL PRODUCTS BRANCH

GEORGIA INSTITUTE OF TECHNOLOGY

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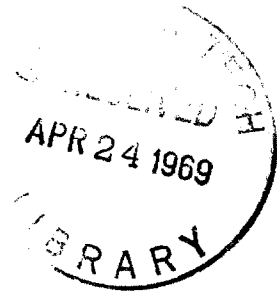
ATLANTA, GEORGIA 30332

April 5, 1965

Division of Highway Planning
State Highway Department of Georgia
Atlanta, Georgia

Attention: Mr. H. H. Huckeba
State Highway Planning Engineer

Subject: Monthly Progress Letter 3, Project A-802
"Hot Melt Traffic Marking Materials"
Contract No. 6401
Covering the Period from March 1 to March 31, 1965



Gentlemen:

Laboratory activities during the past month have been confined almost exclusively to assembly and check-out of apparatus for preparation and application of hot melt compositions for field tests. It is anticipated that trial applications may be made during the current month.

Additional data has been accumulated on cold flow of the several hot melt formulations on hand.

A need for a special impact tester was not anticipated until early project work clarified the physical property requirements of hot melts. A study is being made of available instruments to meet our particular needs. We shall endeavor to acquire this capital item on a Station Equipment Budget.

Respectfully submitted,

W. R. Tooke, Jr. /
Project Director

Approved:

W. H. Burrows, Head
Industrial Products Branch

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GEORGIA INSTITUTE OF TECHNOLOGY

ENGINEERING EXPERIMENT STATION

ATLANTA, GEORGIA 30332

May 6, 1965

Division of Highway Planning
State Highway Department of Georgia
Atlanta, Georgia

Attention: Mr. H. H. Huckeba
State Highway Planning Engineer

Subject: Monthly Progress Letter 4, Project A-802
"Hot Melt Traffic Marking Materials"
Contract No. 6401
Covering the Period from April 1 to April 30, 1965

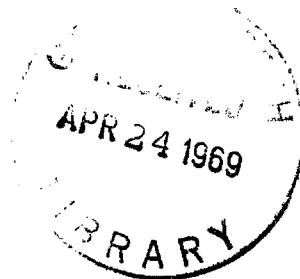
Gentlemen:

Laboratory activities during April have included testing and modification of field application apparatus and preparation of quantities of experimental hot melt compositions for field applications.

The plan for the initial field tests has been enlarged to furnish additional important performance information. As now conceived, all hot melt formulations (2 proprietary and 6 experimental) will be applied as cross stripes at two thicknesses (approximately 3/16" and 3/32") on concrete and asphalt. Additionally, on the concrete priming treatments with a rubber-phenolic primer and with a catalyzed epoxy clear primer will be compared with the unprimed surface. Control paints will consist of the standard alkyd and the chlorinated rubber modified alkyd utilized throughout Project B-210. These paints will be applied at 10, 15, and 20 mils beaded on both concrete and asphalt. The experimental scheme is summarized in the tabulation attached. It will be seen that the total plan contemplates:

8 Hot Melts x 2 Thicknesses x 4 Surfaces	=	64
2 Paints x 3 Thicknesses x 2 Surfaces	=	<u>12</u>
Total Stripes	=	76

The originally planned purpose of obtaining performance data on hot melts having various physical property characteristics continues to be the main objective of this study. Upon further consideration it was decided, however, that now is the appropriate time to evaluate both surface and thickness effects so that the treatment of these variables may be reduced in subsequent tests. Comments and suggestions on these plans would be appreciated. It should be observed that we do not yet have a compositional specification on the epoxy primer. Subject to other suggestions we plan to consult with R. M. suppliers and examine a sample of proprietary epoxy primers as a basis for preparation of a compositional specification.



Monthly Progress Letter 4
Project A-802

Page 2
May 6, 1965

If our preparations are not unexpectedly delayed, the field applications may be scheduled for the last week of May.

Respectfully submitted,

W. R. Tooke, Jr./
Project Director

Approved:

W. H. Burrows, Head
Industrial Products Branch

Attachment

FIELD EXPOSURE TEST PLAN, SERIES I

Materials (10)						
Thickness	-	8 Hot Melts		2 Paints		
		<u>3/16"</u>	<u>3/32"</u>	<u>10 mils</u>	<u>15 mils</u>	<u>20 mils</u>
<u>Concrete</u>						
Unprimed		x	x	x	x	x
Rubber Phenolic		x	x			
Epoxy		x	x			
<u>Asphalt</u>						
Unprimed		x	x	x	x	x

GEORGIA INSTITUTE OF TECHNOLOGY

ENGINEERING EXPERIMENT STATION

ATLANTA, GEORGIA 30332

June 8, 1965



Division of Highway Planning
State Highway Department of Georgia
Atlanta, Georgia

Attention: Mr. H. H. Huckeba
State Highway Planning Engineer

Subject: Monthly Progress Letter 5, Project A-802
"Hot Melt Traffic Marking Materials"
Contract No. 6401
Covering the Period from May 1 to May 31, 1965

Gentlemen:

During May laboratory activities were concentrated upon preparations for field application tests which had been scheduled for the first week in June.

A modification of the field applicator to provide a quick change in application thickness was installed and tested. A number of practice applications were made. It became necessary to increase the capacity of the premelting equipment to accommodate quantities of 3 gallons each of hot melts.

Compositional information could not be obtained on proprietary epoxy primers; however, the characteristics of proprietary samples were studied and duplicated very closely.

In view of the apparent use of non-drying alkyds in proprietary compositions of hot melts, special interest was directed toward formulation of a similar product for inclusion in the present series. A reasonable candidate material was prepared. Thus in addition to two proprietary hot melts, this initial exposure series includes formulations based on non-drying alkyd, S/A copolymer resin, and ethyl cellulose.

Respectfully submitted,

W. R. Tooke, Jr.
Project Director

Approved:

W. H. Burrows, Head
Industrial Products Branch

GEORGIA INSTITUTE OF TECHNOLOGY

ENGINEERING EXPERIMENT STATION

ATLANTA, GEORGIA 30332

July 30, 1965

Division of Highway Planning
State Highway Department of Georgia
Atlanta, Georgia

Attention: Mr. H. H. Huckeba
State Highway Planning Engineer

Subject: Monthly Progress Letter 6, Project A-802
"Hot Melt Traffic Marking Materials"
Contract No. 6401
Covering the Period from July 1 to July 31, 1965



Gentlemen:

During July project activities have been directed towards further rational selections of physical tests to characterize performance capabilities of hot melts. Choices of methods are influenced by the following considerations:

1. Service environment and dynamic requirements.
2. Avoidance of excessive redundancy in characterizing basic physical properties.
3. Use of standard test methods where appropriate.
4. Types of equipment presently available or easily assembled.

The methods that have been selected include the following:

1. Melt consistency, 300° to 475° F, Brookfield RVT, Spindle No. 6, 20 RPM.
2. Density, water displacement.
3. Thermal Coefficient of Expansion, Method 2032*.
4. Flexural Creep, 77° F and 10° F, standard loads.
5. Tensile strength and modulus (stress-strain curves) at 77° F and 10° F, Method 1011.
6. Bond strength, tensile and shear at 77° F and 10° F.

* Federal Specification L-P-406b.

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7. Impact Strength at 77° F and 10° F, Charpy and/or Izod, energy absorbed, maximum load and deformation.
8. Impact Bond Strength at 77° F and 10° F shear and tensile, energy absorbed and maximum load.
9. Wear test using accelerated wear tester, Condition I (heavy abrasive effects).

A detailed justification of the various methods will not be attempted at this time; however, it will be obvious that we are particularly interested in the relations of temperature to strength and modulus characteristics under both static and dynamic (impact) conditions.

Testing work is in progress on the methods excepting the strength characteristics for which acquisition of some special molds and auxiliaries has been necessary.

Respectfully submitted.

W. R. Tooke, Jr.
Project Director

Approved:

W. H. Burrows, Head
Industrial Products Branch

GEORGIA INSTITUTE OF TECHNOLOGY

ENGINEERING EXPERIMENT STATION

ATLANTA, GEORGIA 30332

September 3, 1965



Division of Highway Planning
State Highway Department of Georgia
Atlanta, Georgia

Attention: Mr. H. H. Huckeba
State Highway Planning Engineer

Subject: Monthly Progress Letter 7, Project A-802
"Hot Melt Traffic Marking Materials"
Contract No. 6401
Covering the Period from 1 August to 31 August 1965

Gentlemen:

August project activities have been directed to physical testing problems. Certain modifications of proposed methods have been found necessary for adaptation to available equipment.

1. Static Tensile Testing.

A series of tests performed on equipment owned by Instron Engineering Corporation indicated that reliable tensile stress-strain data cannot be obtained without extensometer equipment which is not presently available to us. As an alternative, we propose to utilize static flexure testing which will yield meaningful force-deflection data on our Instron machine. A simple specimen holder was designed for this purpose.

2. Static Tensile and Shear Bond Strength.

Specimen configuration and testing assemblies were designed. If possible, the same configuration is to be utilized for impact bond tests.

3. Thermal Coefficient of Expansion.

A specimen holder was constructed for this test.

4. Molding Test Specimens.

Much experimentation has been directed to specimen preparation on the premise that the technique should simulate service application conditions, but must also yield highly uniform specimens. Compression

molding techniques were ruled out in favor of open casting processes. Subject to subsequent test findings, we believe that satisfactory uniformity and dimensional precision have been realized in the preparation procedure.

Respectfully submitted,

W. R. Tooke, Jr.
Project Director

Approved:

W. H. Burrows, Head
Industrial Products Branch

GEORGIA INSTITUTE OF TECHNOLOGY

ENGINEERING EXPERIMENT STATION

ATLANTA, GEORGIA 30332

November 3, 1965



Division of Highway Planning
State Highway Department of Georgia
Atlanta, Georgia

Attention: Mr. Emory C. Parrish
State Highway Planning Engineer

Subject: Monthly Progress Letter 8, Project A-802
"Hot Melt Traffic Marking Materials"
Contract No. 6401
Covering the Period from October 1 to October 31, 1965

Gentlemen:

October project activities have continued with major emphasis on physical testing work. Attention was also directed to a determination of appropriate levels of titanium dioxide pigment in hot melts.

1. Static Bond Strengths

Evidence of a detrimental effect of moisture in concrete on bond strengths has been confirmed. The moisture content and tensile bond strengths of unprimed test concrete blocks is as follows:

	<u>Concrete Block Moisture Content, %</u>	<u>Catatherm Tensile Bond Strength, psi</u>
Oven Dried	0	55
Laboratory Equilibrium Moisture	4	50
Saturated Surface Dry	7.5	25

Thus it is seen that equilibrium moisture does not adversely affect bonding as compared with oven dried conditions, but at saturation moisture levels bonding is drastically reduced on unprimed concrete. The effect of priming on moisture sensitivity remains to be determined.

Tensile bond testing of primed (epoxy and Pliogrip) concrete blocks yielded such high bond strengths that cohesive failures of the hot melts predominated; therefore, attention was directed to shear bond tests in an effort to obtain more meaningful data. The shear bond tests have disclosed significant differences among the various types of hot melts when tested both at room temperature and at 0° F. The effect of the low temperature on the unprimed bond of all the hot melts tested is catastrophic. Data on primed systems at 0° F are incomplete but suggest that considerable improvement is effected.

In another preliminary study, unprimed specimens of several types of hot melts have been subjected to one cycle of 0° F temperature, allowed to recover at room temperature for 3 days, and then tested at room temperature. The effect was still catastrophic with the exception of the Calif. Spec. (#26). In this case, the specimen recovered about one-half of its original strength.

2. Titanium Dioxide Pigmentation Level

Initial formulation work had been based on the California Specification which included titanium dioxide in the formulation at a level of approximately 13 per cent by weight of the total formula. This appeared to be an unnecessarily generous loading of opacifying pigmentation, and experiments were initiated to explore reduced loadings. It has been found that as little as one-fourth of the specified concentration may be used without undue loss of opacity and whiteness. Accordingly, future formulation work is being based on loadings of about 3.5 per cent. It is recognized that vehicle color and specified whiteness requirements of a final product may dictate loading variations from this nominal level.

3. Other Activities

The impact testing machine was received in October, and is being adapted for impact tests. In addition to the static bond testing that has been described, specimens have been prepared for static flexure, creep, and coefficient of linear thermal expansion determinations. Utilizing the guidance of the resulting test data on initial formulations, during November attention will be directed towards optimizing compositions for maximum performance potentials.

Respectfully submitted,

W. R. Tooke, Jr.
Project Director

Approved:

W. H. Burrows, Head
Industrial Products Branch

GEORGIA INSTITUTE OF TECHNOLOGY

ENGINEERING EXPERIMENT STATION

ATLANTA, GEORGIA 30332

December 8, 1965



Division of Highway Planning
State Highway Department of Georgia
Atlanta, Georgia

Attention: Mr. Emory C. Parrish
State Highway Planning Engineer

Subject: Monthly Progress Letter 9, Project A-802
"Hot Melt Traffic Marking Materials"
Contract No. 6401
Covering the Period from November 1 through
November 30, 1965

Gentlemen:

November project activities have been directed towards physical testing and formulation with emphasis on the former.

1. Static Bond Strengths

Moisture content and priming effects have been investigated further. Pliogrip was found to be highly effective in preventing loss of bond to damp concrete. Epoxy was relatively ineffective for this purpose.

Low temperature effects on unprimed bonds were found to be catastrophic. Both types of primer are effective in promoting low temperature bonds to acceptable levels.

Additives to the hot melt composition have been found to enhance the unprimed bond at both room temperature and 0°F. This approach appears to be highly satisfactory.

2. Static Flexural Strengths

Flexural strengths of formulations of immediate interest were determined. Values of test formulations were competitive with proprietary items. Unfortunately, flexural modulus could not be satisfactorily determined during these tests. Only room temperature tests have been run to date.

3. Flexural Creep

Formulations have been retested using standard $\frac{1}{2} \times \frac{1}{2}$ inch cross-section specimens (same as flexural strength) at room temperature. An effort is being made to obtain useful modulus data on this apparatus.

4. Impact Testing

Modifications of the impact tester were required to reduce the energy levels to obtain useful data.

5. Thermal Coefficient

Some data has been obtained (values in the range of 3 to $4 \times 10^{-4}/^{\circ}\text{F}$) but the technique requires refinement.

6. Formulation

Systematic studies of selected additives in a standard alkyd-base (HM #28) formulation have been started.

Respectfully submitted,

W. R. Tooke, Jr.
Project Director

Approved:

W. H. Burrows, Head
Industrial Products Branch

GEORGIA INSTITUTE OF TECHNOLOGY

ENGINEERING EXPERIMENT STATION

ATLANTA, GEORGIA 30332

February 2, 1966



Division of Highway Planning
State Highway Department of Georgia
Atlanta, Georgia

Attention: Mr. Emory C. Parrish
State Highway Planning Engineer

Subject: Monthly Progress Letter 10, Project A-802
"Hot Melt Traffic Marking Materials"
Contract No. 6401
Covering the Period from January 1 to January 31, 1966

Gentlemen:

January project activities have continued with the work of systematic formulation, testing, and interpretation. Further analysis of the formulation and physical property data presented in Quarterly Progress Report No. 4 dated January 10, 1966 has revealed the necessity for re-evaluations of some of the test methods and of the vehicle formulation rationale.

More specifically, the hot melt consistency determination method is being re-examined. We suspect that some of the filler may settle out during a determination and cause misleading data. Several test variables are being studied, and we are adopting routine consistency characterizations of clear (unfilled) binders to preclude any possibility of misinterpretation of the consistency effects of experimental binder constituents.

Statistical control of the static shear bond test is not satisfactory for purposes of formulation guidance, although it has been most valuable in indicating certain trends. Modifications of this test are under study.

The overall results to date suggest the desirability of taking two separate approaches to hot melt formulation. Our findings have indicated that with proper priming any reasonable hot melt composition will attain good adhesion. Without priming, it appears necessary to trade-off certain desirable physical properties to achieve good adhesion. We propose to investigate a range of characteristics which will include both extremes. An ultimate choice (primer or no primer) would depend upon the increment of durability attainable by priming.

Our current formulation work is aimed at attainment of this range of characteristics in a set of compositions that will comprise our second series of highway cross stripe tests to be placed within the next few months.

Respectfully submitted,

W. R. Tooke, Jr. /
Project Director

Approved:

W. H. Burrows, Head
Industrial Products Branch



GEORGIA INSTITUTE OF TECHNOLOGY

ENGINEERING EXPERIMENT STATION

ATLANTA, GEORGIA 30332

March 7, 1966

Division of Highway Planning
State Highway Department of Georgia
Atlanta, Georgia

Attention: Mr. Emory C. Parrish
State Highway Planning Engineer

Subject: Monthly Progress Letter 11, Project A-802
"Hot Melt Traffic Marking Materials"
Contract No. 6401
Covering the Period from 1 February to 28 February 1966

Gentlemen:

Within the last few days, an informal observation of the hot melt test stripes on I85 at the Lenox Road Bridge gave evidence that chipping failure has advanced on a few of the formulations. The effect seems to be confined mainly to the unprimed stripes on concrete. With these few exceptions, the hot melts appear to be in almost perfect condition after nine months of very heavy traffic, including the freeze-thaw cycling of the winter season. In contrast, the control paints are at about an ASTM rating of 5 or less; thus, they exhibit now a clear inferiority to the hot melts in durability. A formal inspection to include night visibility and photographs is scheduled for next week.

These field results are indeed encouraging, but our laboratory findings indicate that one or more of the properties of each of the field formulations can and probably should be improved to attain maximum durability.

During the last month a means was found to promote improved adhesion without excessive creep or undue loss of toughness. Thus we feel that we are approaching a satisfactory candidate formulation for use without a primer. Our efforts to attain the maximum of toughness in a formulation designed to yield ultimate durability with priming have been frustrated by what appears to be an inherent characteristic of thermoplastics. Briefly,

toughness ~ molecular weight ~ viscosity

Some of our best results (toughness) have been achieved with ethyl cellulose as the polymeric constituent, but we are concerned with evidence that its thermal stability is marginal. From the chemical viewpoint, one observes that polymers which do not hydrogen bond, i.e., vinyl types and hydrocarbons, require higher molecular weights to attain high tenacity than do hydrogen

bonding types, i.e., cellulose, polyesters, polyamides, etc. Accordingly, we have tended to concentrate interest on the latter. Locating suitable compatible resin extenders and fluxing materials has been the major difficulty. Progress is being made, but ideas or suggestions in this area would be most gratefully received.

We are proceeding with an overhaul and minor design improvements to our field application equipment, anticipating placement of a final series of test stripes this spring.

Respectfully submitted,

W. R. Tooke, *Jr.*
Project Director

Approved:

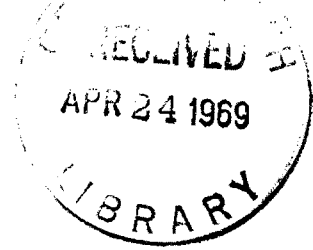
W. H. Burrows, Head
Industrial Products Branch

GEORGIA INSTITUTE OF TECHNOLOGY

ENGINEERING EXPERIMENT STATION

ATLANTA, GEORGIA 30332

May 5, 1966



Division of Highway Planning
State Highway Department of Georgia
Atlanta, Georgia

Attention: Mr. Emory C. Parrish
State Highway Planning Engineer

Subject: Monthly Progress Letter 12, Project A-802
"Hot Melt Traffic Marking Materials"
Contract No. 6401
Covering the Period from April 1 to April 30, 1966

Gentlemen:

During the past month preparations have been intensified for application of a second series of hot melt formulations on the highway test site.

Modifications of the hot melt applicator have been completed to include a "corrugator wheel" which trails the applicator to apply a surface configuration to the stripe which will enhance the wet night visibility. The operation of the corrugator wheel has not been checked out yet; however, we have applied corrugations manually to test stripes placed on our parking lot. A 1 inch diameter steel rod was used to impress transverse corrugations at intervals of about 4 inches. The resulting pattern was found to yield very good retroreflection when illuminated and observed at very low angles. The retroreflection was not appreciably affected when the stripe was drenched with water. A Record of Invention was filed on this concept on April 12, 1966, and a patent search is in progress. We are planning to make comparative evaluations of the corrugated hot melt, the conventional hot melt and conventional paint stripes in the upcoming field tests. These evaluations will require longitudinal (down the road) placement of stripes rather than the transverse placements heretofore used.

In this connection, we have found it necessary to make some design improvements in our paint applicator, including the installation of a better bead distributor. This will permit us to make more critical comparative observations of the night visibility characteristics of the three types of striping.

The area of laboratory investigation of hot melt vehicles has been narrowed for the present to an intensive systematic study of a single polyamide polymer in combination with one plasticizer and one extender resin. This system exhibits a combination of physical properties greatly superior to anything studied previously.

Our forthcoming highway placements will be devoted largely to selected variations of this polyamide system. We are also planning to include an alkyd system which has previously demonstrated satisfactory performance and is particularly economical in raw materials costs. Finally, we propose to repeat the placements of the California Specifications, one proprietary, and the Georgia Specification paint as controls and references.

For the present, we have not attempted to make formal data reductions of our 9 months highway observations, but we are setting up for an analysis of variance on the one year observations to be taken in June. It is abundantly clear now that the hot melt compositions presently on the road are much more durable than our best paints, and on this basis we may anticipate conservatively that integrity failures might not be recorded on some formulations for as long as three or four years. This period would go considerably beyond the present project termination date even for the initial placements in June 1965 and certainly for those to be placed in about another month.

On the other hand, it appears probable that night visibility of hot melts cannot be maintained at the desired levels during the integrity life of the stripe. This may demand that some means be found to economically rejuvenate night visibility of existing stripes. This would pose a new problem that is beyond the scope of the present project.

Project A-802 is currently "on schedule" with the possible exception of the second series of highway placements which have been deferred to enable us to complete more laboratory work on polyamide systems. We anticipate that existing project objectives will be completed within the contract period. These objectives do not include evaluation of highway performance beyond about two years, nor the above mentioned night visibility rejuvenation. In addition, the corrugation technique can receive only exploratory study within the present project scope. Accordingly, recommendations for new project work based on these findings will be made in the near future.

Respectfully submitted,

W. R. Tooke, Jr.
Project Director

Approved:

Frédéric Bellinger, Chief
Chemical Sciences and Materials Division

GEORGIA INSTITUTE OF TECHNOLOGY

ENGINEERING EXPERIMENT STATION

ATLANTA, GEORGIA 30332

June 2, 1966



Division of Highway Planning
State Highway Department of Georgia
Atlanta, Georgia

Attention: Mr. Emory C. Parrish
State Highway Planning Engineer

Subject: Monthly Progress Letter 13, Project A-802
"Hot Melt Traffic Marking Materials"
Contract No. 6401
Covering the Period from May 1 to May 31, 1966

Gentlemen:

A peak rate-of-effort was applied to this project during May. Final decisions were made on formulations for the final highway test exposures. Application equipment was modified. Large quantities of hot melts were prepared, and the field applications were conducted on May 30 and 31.

Details will be presented in the next quarterly report; however, the highway placements may be summarized as follows:

Materials:

	<u>Formulation No.</u>
Georgia Spec. No. 4 Traffic Paint, White	159
Catatherm Hot Melt	164
California Spec. Hot Melt	160
Alkyd Type, Low	162
Alkyd Type, High	163
Polyamide Type, Medium	155
Polyamide Type, High	157
Polyamide Type, Low	158

Application Thicknesses:

Hot Melts	125 mils (1/8 in.)
Paint	15 mils

Substrates:

P/C Concrete
Asphalt

At the primary testing site on I-85 at the Lenox Road Bridge, the entire set of materials was applied as transverse stripes on both concrete and asphalt. At a secondary site on I-85 between North Druid Hills Road and Clairmont Road, a series of longitudinal stripes was applied using only Paint # 159 and Hot Melt # 155. Finally, a set of three stripes was applied on the parking lot west of the Architecture Building at Georgia Tech. This latter installation was designed to permit night visibility observations without traffic interference.

On the same days of these new placements we also made observations and photographs of the original placements at one year's weathering. Additional cracking and flaking is evident since the nine months' observations, but a number of stripes continue to exhibit virtually perfect integrity. A further deterioration of night visibility was noted in most cases.

Future work on the project will involve continued field observations and correlation with laboratory testing to provide the basis for specifications of hot melt compositions and properties.

Respectfully submitted,

W. R. Tooke, Jr.
Project Director

Approved:

Frederick Bellinger, Chief
Chemical Sciences and Materials Division

GEORGIA INSTITUTE OF TECHNOLOGY

ENGINEERING EXPERIMENT STATION

ATLANTA, GEORGIA 30332

August 9, 1966

Division of Highway Planning
State Highway Department of Georgia
Atlanta, Georgia

Attention: Mr. Emory C. Parrish
State Highway Planning Engineer

Subject: Monthly Progress Letter 14, Project A-802
"Hot Melt Traffic Marking Materials"
Contract Number 6401
Covering the Period from July 1 to July 31 1966



Gentlemen:

During July complete laboratory characterization testing was performed on retained samples of the Hot Melts of Highway Series II (6/1/66). Subsequently, we will undertake to correlate the laboratory tests with field performance. We have also continued to examine certain conditions which appear to affect laboratory test results significantly. With some hot melt compositions, for example, the time-temperature history has a large effect on the physical properties, even though the appearance of the material may be only negligibly affected. Possibly in some cases more volatile plasticizing material is driven off by prolonged heating increased brittleness; in others, degradation may yield products which increase the plasticity of the composition. Undoubtedly, all types of compositions undergo some property changes.

Laboratory work has also been started on special test procedures for hot melt specifications. A major effort is being made to define performance requirements fully by laboratory procedures, and avoid, if possible, the necessity for compositional specifications.

On the basis of studies pursued under this project, a proposal has been drafted on "Demonstration and Maintenances of High Visibility Hot Melt Traffic Marking Materials and Methods."

This proposal is intended to provide a full-scale practical demonstration of the wet night visibility potentials of textured hot melt traffic marking.

Respectfully submitted,

W. R. Tooke, Jr.
Project Director

Approved:

Frederick Bellinger, Chief
Chemical Sciences and Materials Division

A-802

GEORGIA INSTITUTE OF TECHNOLOGY

ENGINEERING EXPERIMENT STATION

ATLANTA, GEORGIA 30332

September 8, 1966



State Highway Department of Georgia
2 Capitol Square, S. W.
Atlanta, Georgia 30302

Attention: Mr. Leland S. Veal
State Highway Planning Engineer

Subject: Monthly Progress Letter 15, Project A-802
"Hot Melt Traffic Marking Materials"
Contract Number 6401
Covering the Period from August 1 to August 31, 1966

Gentlemen:

Project activity during August was directed primarily towards the development of suitable laboratory testing procedures for acceptance testing of hot melt compositions. A corollary study involved an evaluation of effects of prolonged heating on hot melt properties. This study has resulted in the adoption of a heat aging treatment of hot melts as the initial step in the specification testing routine. Adequate thermal stability is an essential property of practical hot melts.

Water resistance and freeze-thaw integrity are the main environmental factors that are being studied in the development of specification tests. Among representative compositions examined water resistance is very good, but freeze-thaw cycling is extremely destructive to bond strengths of some hot melts. Minimum performance requirements are being established.

Until recently, our laboratory testing has not yielded predictions of field application characteristics of various formulations. Large variations in application characteristics were observed within a narrow range of formulation consistencies. Furthermore, better application does not necessarily accompany lower consistency. Consistency is, of course, only one of several pertinent rheological parameters. We suspected that an elastic flow characteristic was responsible for poor application in some cases. Examination of creep curves tended to confirm this suspicion. Poor application properties were found to accompany large initial elastic deformations on the creep curves. Regrettably (from the application point-of-view), elastic deformation appears to be associated with desirable performance characteristics. However, formulation dilemmas are inevitable and must always be managed by compromises. The generally satisfactory performance of field tests to date indicates that application properties have not been favored excessively. Furthermore, the anticipated performance superiorities of several field-applied formulations that exhibited marginal application characteristics have not yet been demonstrated.

During the month of August all placements of hot melt test stripes on the asphalt surfaces of Interstate 85 were destroyed by unanticipated repaving operations. This included a set of cross stripes placed in June 1965 and another set placed in June 1966. This represents a regrettable loss of much painstaking work and valuable technical information. The loss is partially compensated by the fact that the test stripes are all replicated on concrete surfaces, and the findings at one year's weathering indicate that durability on asphalt is superior. Thus, the concrete tests should indicate minimum performance capabilities. Monitoring of these stripes on concrete will be continued.

Respectfully submitted,

W. R. Tooke, Jr.
Project Director

Approved:

Frederick Bellinger, Chief
Chemical Sciences and Materials Division

GEORGIA INSTITUTE OF TECHNOLOGY

ENGINEERING EXPERIMENT STATION

ATLANTA, GEORGIA 30332

November 8, 1966

State Highway Department of Georgia
2 Capitol Square, S.W.
Atlanta, Georgia 30302

Attention: Mr. Leland S. Veal
State Highway Planning Engineer

Subject: Monthly Progress Letter 16, Project A-802
"Hot Melt Traffic Marking Materials"
Contract No. 6401
Covering the Period from October 1 to October 31, 1966

Gentlemen:

Specification test development work has been continued with emphasis on studies of sample conditioning and cycling environments as a means of assessing performance limitations. Testing procedures are being simplified, where possible, to provide only the necessary acceptance data, and to omit details that are relevant only to research work.

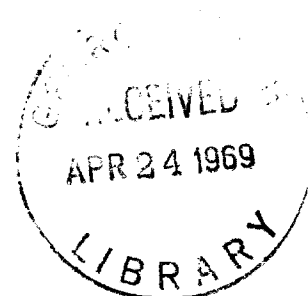
A critical review has been initiated of all of the experimental studies undertaken on this project. The findings will be summarized to provide concise guidance for future formulation work. This will represent one phase of the final project report.

Respectfully submitted:

W. R. Tooke, Jr.
Project Director

Approved:

Frederick Bellinger, Chief
Chemical Sciences and Materials Division



A-802

GEORGIA INSTITUTE OF TECHNOLOGY

ENGINEERING EXPERIMENT STATION

ATLANTA, GEORGIA 30332

December 8, 1966

State Highway Department of Georgia
2 Capitol Square, S.W.
Atlanta, Georgia 30302

Attention: Mr. Leland S. Veal
State Highway Planning Engineer

Subject: Monthly Progress Letter 17, Project A-802
"Hot Melt Traffic Marking Materials"
Contract No. 6401
Covering the Period from November 1 to November 30, 1966

Gentlemen:

A proposal has been prepared on "Wet Night Visibility Study" and was forwarded to the State Highway Department of Georgia on November 1, 1966. This proposal describes full-scale development and evaluation of a concept that has been studied and reported under this project. (See Project No. A-802, Monthly Progress Letter No. 12, May 5, 1966, and subsequent progress reports for details on "Corrugated Hot Melt Traffic Stripes.") Current observations of corrugated cross-stripe highway placements made last June, indicate continued satisfactory performance.

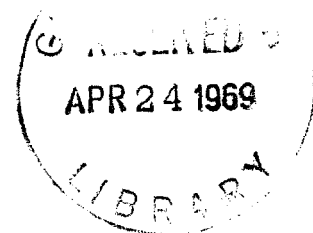
The initially planned program of laboratory formulation and physical testing was concluded in November. Primary attention is now being given to a review and analysis of findings in support of a final report on formulation technology, testing, and specifications. Future laboratory work will be confined to closing any gaps that may be found within the existing experimental program. Special attention will be given to verifications of useful formulation principles.

Respectfully submitted:

W. R. Tooke, Jr.
Project Director

Approved:

Frederick Bellinger, Chief
Chemical Sciences and Materials Division



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GEORGIA INSTITUTE OF TECHNOLOGY

ENGINEERING EXPERIMENT STATION

ATLANTA, GEORGIA 30332

February 8, 1967

State Highway Department of Georgia
2 Capitol Square, S.W.
Atlanta, Georgia 30302

Attention: Mr. Leland S. Veal
State Highway Planning Engineer

Subject: Monthly Progress Letter 18, Project A-802
"Hot Melt Traffic Marking Materials"
Contract No. 6401
Covering the Period from January 1 to January 31, 1967

Gentlemen:

On January 31, 1967 all highway cross-stripes were examined for integrity and night visibility. The exposure period for Series I was 20 months, and Series II was 8 months. Since detailed data reductions will be developed for the final report, only a few observations of special interest will be mentioned at this time.

Among the Series I stripes, a number remain with virtually perfect integrity. These are mostly full thickness stripes, epoxy primed, and are confined to formulations based on the California Specification, on ethyl cellulose, and on alkyd. Night visibilities have dropped from the levels reported at one year, and the best values remaining are Permaline at 13, California Specification ($\frac{1}{2}$ Pliolite) at 10, and Alkyd at 10. An incidence of heavy cracking in two other formulations in the series has contributed to our understanding of physical property limitations.

Series II has exhibited catastrophic adhesion failures of unprimed polyamide formulations (H.M. # 155, # 157, # 158). This was unexpected in view of superior adhesion observed in laboratory tests. Further study of this problem has been undertaken.

It is important that we gain an understanding of the causes of performance failures of hot melts, and we shall continue to direct special attention to this subject. At the same time, we would emphasize the more positive fact that practical hot melt stripes of defined compositions have clearly demonstrated great superiority to high quality traffic paints within this continuing program.

Respectfully submitted,

W. R. Tooke, Jr.
Project Director

Approved:

Frederick Bellinger, Chief
Chemical Sciences and Materials Division

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A-802

GEORGIA INSTITUTE OF TECHNOLOGY

ENGINEERING EXPERIMENT STATION

ATLANTA, GEORGIA 30332

March 6, 1967

State Highway Department of Georgia
2 Capitol Square, S.W.
Atlanta, Georgia 30302

Attention: Mr. Leland S. Veal
State Highway Planning Engineer

Subject: Monthly Progress Letter 19, Project A-802
"Hot Melt Traffic Marking Materials"
Contract No. 6401
Covering the Period from February 1 to February 28, 1967

Gentlemen:

Project work during this reporting period has consisted primarily of organization and analysis of a large volume of laboratory and field data, and drafting work on the final report. Some laboratory testing has been performed as needed to complete data for reporting.

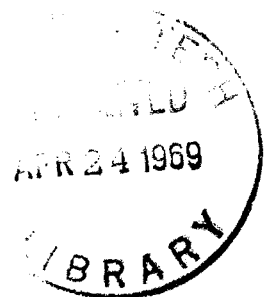
Future work on this project will be pursued at a low rate-of-effort, and shall be confined primarily to preparation of the final report. The work will be concluded as soon as feasible following highway test observations next June.

Respectfully submitted,

W. R. Tooke, Jr.
Project Director

Approved:

Frederick Bellinger, Chief
Chemical Sciences and Materials Division



GEORGIA INSTITUTE OF TECHNOLOGY

ENGINEERING EXPERIMENT STATION

ATLANTA, GEORGIA 30332

May 9, 1967

[REDACTED]

State Highway Department of Georgia
2 Capitol Square, S.W.
Atlanta, Georgia 30302

Attention: Mr. Leland S. Veal
State Highway Planning Engineer

Subject: Monthly Progress Letter 20, Project A-802
"Hot Melt Traffic Marking Materials"
Contract No. 6401
Covering the Period from April 1 to April 30, 1967

Gentlemen:

During April project activities were confined to drafting work on the final report and on specifications for hot melt traffic marking material. Specifications for a primer were submitted with Quarterly Report No. 9, April 6, 1967.

As noted previously, the final report will be concluded as rapidly as possible following June observations of highway stripes.

Respectfully submitted,

W. R. Tooke, Jr.
Project Director

Approved:

Frederick Bellinger, Chief
Chemical Sciences and Materials Division

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GEORGIA INSTITUTE OF TECHNOLOGY

ENGINEERING EXPERIMENT STATION

ATLANTA, GEORGIA 30332

June 2, 1967

State Highway Department of Georgia
2 Capitol Square, S.W.
Atlanta, Georgia 30302

Attention: Mr. Leland S. Veal
State Highway Planning Engineer

Subject: Monthly Progress Letter 21, Project A-802
"Hot Melt Traffic Marking Materials"
Contract No. 6401
Covering the Period from May 1 to May 31, 1967



Gentlemen:

Drafting of the final report was continued together with the development of hot melt specifications. As the specifications now stand, we describe two types of hot melt. Type I is intended mainly for new work or large-scale application and requires use of primer. Type II is intended mainly for repairs or small-scale application, and primer is recommended but not required. This approach is indicated at present because we have found it necessary to "trade-off" other desirable properties in order to gain good adhesion on unprimed concrete (Type II). Where priming is required, this "trade-off" is unnecessary (Type I), and the hot melt exhibits superior properties.

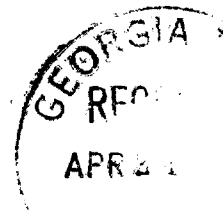
Final observations of highway stripes have been scheduled for June 14.

Respectfully submitted,

W. R. Tooke, Jr.
Project Director

Approved:

Frederick Bellinger, Chief
Chemical Sciences and Materials Division



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ENGINEERING EXPERIMENT STATION

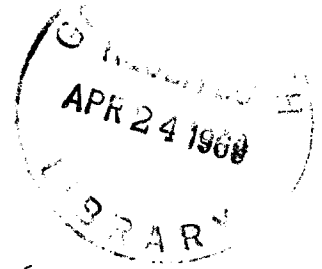
ATLANTA, GEORGIA 30332

August 10, 1967

State Highway Department of Georgia
2 Capitol Square, S.W.
Atlanta, Georgia 30302

Attention: Mr. Leland S. Veal
State Highway Planning Engineer

Subject: Monthly Progress Letter 22, Project A-802
"Hot Melt Traffic Marking Materials"
Contract No. 6401
Covering the Period from July 1 through July 31, 1967




Gentlemen:

An analysis-of-variance study is being run on the data from the two series of highway cross stripe tests. The results will be included in the final report now in preparation. The draft of this report is nearly complete.

Specifications for "Thermoplastic Traffic Line Material" have been developed concurrently, and the present draft of these specifications is attached. The specifications are based entirely on physical properties and test performance characteristics of the material and impose no compositional requirements whatsoever.

Respectfully submitted,

W. R. Tooke, Jr. 
Project Director

Enclosure: Specifications for "Thermoplastic Traffic Line Material"

Approved:

Frédéric Bellinger, Chief
Chemical Sciences and Materials Division

STATE HIGHWAY DEPARTMENT OF GEORGIA
TENTATIVE SPECIFICATION
FOR
THERMOPLASTIC TRAFFIC STRIPE

General

This tentative specification was developed under Research Project No. A-802 (Contract No. 6401) based upon experimental findings and currently available technical information. It is requested that recommendations for changes be directed to the Engineer of Materials and Tests of the State Highway Department of Georgia.

No prior specifications have been issued covering the product and application described hereunder.

Scope and Classification

Scope - This is a performance specification for thermoplastic traffic striping material which is applied while molten with immediate drop on application of glass beads. Upon cooling to pavement temperature the material solidifies to form a durable retroreflective stripe.

Classification - The thermoplastic material shall be of the following two types as specified in the invitation for bids, contract, or order.

Type I - White

Type II - Yellow

Requirements

Materials - The manufacturer is granted wide latitude in selecting materials and in formulating to meet the performance requirements of this specification.

Conditioning Requirements - All specimens of hot melt material selected for testing shall first be heat aged as specified below. In addition, a water-freeze cycle treatment is specified for use with tensile bond strength and impact tests.

Heat Aging Treatment

Temperature	-	400 \pm 10° F
Time	-	4 hours
Agitation	-	Slow, continuous, approx. 250 RPM

The material for treatment is melted in a heated, 1 quart stainless steel beaker with a propeller agitator that is set close to the bottom. The beaker should be at least half filled with molten material. Timing begins when the specified temperature is reached. Test specimens should be prepared immediately following conditioning.

Water-Freeze Cycle Treatment

Samples are placed in a water bath maintained at 77° F for 18 hours. The samples are then transferred to a freezer maintained at 0° F for 3 hours. The samples to be tested at 0° F are tested as soon as they are removed from the freezer. The samples to be tested at 77° F are returned to the water bath for 2 hours and then tested.

Performance Requirements - The performance requirements of both Type I and Type II materials shall be identical unless otherwise specified.

Consistency

Consistency of hot melts is measured using a Brookfield viscometer, Model RTV, 20 rpm, and an oil bath maintained at 350° F. The sample of approximately 800 g is placed in a Size 303 can, heated to a uniform temperature slightly above 350° F and placed in the oil bath. A Brookfield spindle is warmed and then placed in the sample and moved around to bring it to the temperature of the hot melt and release any air trapped under it before attaching it to the viscometer. The reading is taken when the temperature of the hot melt has reached 350° F. The sample should be carefully stirred just prior to taking the reading to insure uniformity. The consistency shall be no more than 35,000 cps, nor less than 2,000 cps.

Flexural Creep Test

The flexural creep of a hot melt is obtained by measuring the sagging displacement of a 1/2 x 1/2 x 6 in. bar of hot melt which is suspended over supports 4 inches apart, and a load of 3550 grams applied to the center of the bar. The sagging is measured by a dial micrometer.

The mils of sagging are plotted against time on linear graph paper and the slope at one minute is taken as the flexural creep. This slope shall not exceed 30 in./in./min. x 10⁻³.

Static Indentation Test

The static indentation is obtained by measuring the diameter of the impression left by a 3/4 in. diameter steel ball loaded to 200 grams for 24 hours on a hot melt disk prepared as described in the Falling Ball Impact Test. The measured diameter of indent should not exceed 200 mils or 0.20 of an inch.

Tensile Bond Strength

Specimens for the tensile bond strength of a hot melt are obtained by cementing two 3 x 5 x 3/4 in. concrete blocks together with a 2 x 2 x 1/4 in. layer of hot melt at 350° F in such a manner that the long dimensions of the block are at 90° to each other. Before the cementing operation, the faces of the concrete blocks are sandblasted to remove laitance. The samples are subjected to the water-freeze cycle as a part of the testing procedure. Specimens are tested in a hydraulic press for tensile bond strength. (The sample should be placed in the press in such a manner that the only forces on the bond are tensile forces.) The tensile bond strengths shall be an average of 5 tested specimens at each temperature and the averages shall equal or exceed 15 psi at 77° F and 50 psi at 0° F.

Falling Ball Impact Test

Prepare 10 disks from heat-aged hot melt which are 0.25 ± 0.025 in. thick and approximately 2 in. in diameter.* These disks should not vary more than $\pm 5\%$ in weight. The disks are subjected to the water-freeze cycle as a part of the testing procedure.

A disk is placed on an anvil which is a 3 pound block of brass 2 in. in diameter with the top covered by a 1/16 in. layer of rubber.

A 1/2 in. diameter steel ball is dropped onto the disk from a position one foot above and centered on the disk. Failure is considered to have occurred when a crack appears in the sample. Four of the five disks must withstand three drops of the steel ball without failure for the material to pass.

Reflectance, Type I, White

The daylight luminous reflectance of Type I, after cooling to 77° F, shall not be less than 74% relative to magnesium oxide when tested according to ASTM E 97.

Color, Type II, Yellow

The color of Type II, after cooling to 77° F, shall match Color No. 1315 of Federal Specification TT-C-595.

Notes on Application

The thermoplastic traffic striping is intended to be used with a specified primer on dry, clean concrete surfaces. On application to asphalt surfaces, no primer is required. The application should be made at temperatures no lower than 65° F.

* Aluminum Foil Cups, Fisher No. 8-732, are satisfactory for molds.

Sampling and Testing

Samples shall accompany each bid. Each sample shall consist of a 50 lb. standard package of material. The right is reserved to inspect and sample all lots submitted for use and to inspect the manufacturing process.

Packaging

The thermoplastic traffic striping material shall be packaged in suitable containers to which it will not adhere during shipment or storage. The blocks of cast thermoplastic material shall be approximately 12 x 35-1/2 x 2 inches and shall weigh approximately 50 pounds. Each container label shall designate the color, manufacturer's name, batch number, and date of manufacture.

Certified Data Report

The manufacturer shall submit with his bid a certified test data report. It shall contain the actual test data obtained by the manufacturer in determining compliance with the physical tests required by these specifications. In addition, the manufacturer shall further certify that if awarded the contract the material furnished will be identical with that of the sample submitted. This information shall be held as confidential.

GEORGIA INSTITUTE OF TECHNOLOGY

ENGINEERING EXPERIMENT STATION

ATLANTA, GEORGIA 30332

March 10, 1965



Division of Highway Planning
State Highway Department of Georgia
Atlanta, Georgia

Attention: Mr. H. H. Huckeba
State Highway Planning Engineer

Subject: Quarterly Progress Report No. 1, Project A-802
"Hot Melt Traffic Marking Materials"
Contract No. 6401, Covering the period from
1 December 1964 to 28 February 1965.

Gentlemen:

Project activities for the three months' period have been conducted in the following areas:

- I. State of the Art Survey
- II. Equipment Development
 - A. Preparation and Application Equipment
 - B. Laboratory Testing Devices
- III. Theoretical Stress Analysis
- IV. Formulation Studies
 - A. Proprietary Compositions
 - B. Experimental Compositions
 - C. Physical Property Data
- V. Future Plans

Additional details of these activities follow:

REVIEW
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I. State of the Art Survey

A combined literature and patent search was completed with particular emphasis on locating recent patents covering hot melt traffic marking materials and equipment. The patent search by selected classes, and sub-classes of the Patent Office was not very productive since the class headings are organized according to chemical composition rather than by use. Chemical Abstracts indices disclosed several new patents and publications of interest. The following items are cited as being particularly pertinent to the present study:

U.S. 3,036,928 (5/29/62) Poole, "Retroreflective Composition and Method of Applying Same."

This recent patent discloses hot melt compositions very similar to those currently under investigation. In addition, this patent cites compositions based on non-drying alkyds as the primary resin. It should be noted that the claims of this patent are very broad.

U.S. 3,020,252 (2/6/62) Hancock, "Road Marker Materials and Methods of Making Same."

Mixtures of sulfur, Thiokol and wood rosin are claimed in this patent.

Hancock, C. Kinney, I.E.C. 46, No. 11, pp. 2431-35, November, 1954.
"Plasticized Sulfur Compositions for Traffic Marking."

Discussion of details of developmental work on sulfur compositions is covered in this paper.

Swiss Patent No. 301,822 (12/1/54) "Plastic Moss for Road Marking."

This patent discloses hot melt compositions based on a rosin vehicle plasticized with "vaseline oil."

U.S. 3,018,704 Searight-Cataphate (1/30/62) "Stripe Applicator for Pavements."

This patent discloses an applicator which is currently in widespread commercial use.

A number of other publications and patents of secondary interest have been reviewed.

Much valuable information has been received through personal communications with Mr. Herbert A. Rooney, Senior Chemical Testing Engineer of the

California Division of Highways. This information includes copies of experimental compositional specifications for hot melt traffic marking material.

A "Summary of Experimental Installations of Thermoplastic Pavement Striping Materials," dated April 13, 1964 compiled by the Bureau of Public Roads Office of Research and Development was supplied to us by the Georgia State Highway Department. In view of the extensive information included in this summary, we deferred a plan to circulate a questionnaire seeking similar information.

In lieu of the questionnaire, we have made visits with officials and operating personnel of the Traffic Department of the City of Atlanta to discuss hot melt performance and to inspect equipment and application procedures. We also inspected equipment and procedures during a recent large scale contract test application for the Georgia State Highway Department in the downtown connector area of the Atlanta Expressway System.

On the basis of the foregoing studies and inspections, we feel that an adequate appreciation of the current "State of the Art" has been acquired, subject, of course, to continuing developments.

II. Equipment Development

A. Preparation and Application Equipment

Laboratory Applicator. A small hand applicator was constructed for lab-scale application of hot melts to test discs for accelerated wear test and other laboratory evaluations. Uniform films of controlled thickness have been applied with this device.

Field Applicator. It was determined that commercially available application equipment would not lend itself to our requirements for rapid field application of a number of different compositions in relatively small experimental quantities for cross-stripe tests. Accordingly, a field applicator was designed and constructed for this experimental work. Figure 1 shows this applicator complete except for installation of infra-red heating elements. A beading device is included on the applicator carriage. The operating principles of the applicator are based on proved commercial equipment; therefore, few mechanical problems are anticipated in connection with its operation.

Premelter. This unit was designed to service the field applicator with up to four separate one-gallon samples of pre-melted material. It consists essentially of a four-place gas stove with temperature controls. Gas is supplied from a 20 pound L.P. gas bottle. This equipment will also serve for laboratory pre-melting and compounding of quantities of material necessary for field experiments. All necessary elements for the premelter have been ordered.



Figure 1. Experimental Hot-melt Stripe Applicator.

Compounding Equipment. Small laboratory quantities of hot melts have been compounded in tin cans on a hot plate with hand spatula agitation for mixing and dispersion. This arrangement is only marginally satisfactory with small quantities, and would be completely unsatisfactory for one-gallon batches. An assessment of the heating and agitation needs has been made from the preliminary experiments, and suitable types of compounding equipment are under consideration.

B. Laboratory Testing Devices

Some modifications in the original conceptions of laboratory testing requirements have been indicated on the basis of observed properties of composite vehicles and compounded hot melt compositions. Much initial attention has been directed to melt viscosity-temperature relations for both vehicles and compounds. Determinations with the Brookfield Viscometer have appeared adequate for characterizing application properties.

Cold Flow and Creep. Slow responses of compounds to constant moderate loads at representative temperatures appears to be an important property relating to the stability and stress relief characteristics of applied materials. A simple jig has been constructed to perform flexural creep tests.

Impact Testing. At the other (high) extreme of loading rates, impact tests provide an indication of direct resistance to traffic or tire impact effects. Some preliminary work has been done with a simple falling weight impact test on coated surfaces--observing impact depths and cracking effects. To obtain more fundamental impact data, plans are being developed to adapt an Izod type pendulum test to measure maximum loading and energy absorption of the coating-substrate bond in both shear and tension.

Other Physical Tests. A temperature gradient bar apparatus is being adapted for measurements of softening points. The use of a brittle temperature test, as such, was deferred since the same information would be implicit in low temperature impact tests. Determinations of coefficients of expansion have been deferred until relevance is clearly indicated.

III. Theoretical Stress Analysis

When a layer of thermoplastic is applied to Portland or bituminous concrete, either tensile or compressive axial stresses may occur in the plastic itself, and some shear stress may occur at the bonded interface between the plastic and concrete. The stresses might be considered to come from two sources: (a) cooling of the thermoplastic from its liquid state at 400° F to the range of environmental temperatures, (b) subsequent environmental heating and cooling of the bonded materials within a range of about 150° to 0° F.

At high temperatures, however, thermoplastics flow quite readily and thus stress relieve themselves. Effect "a" and effect "b", in the higher temperature ranges, can therefore be considered negligible; the real range of interest in heating and cooling is probably in the range of about 50° F downward, especially in the region where the plastic exhibits a relatively constant relation of stress to strain. This is the region near and below the glass point of the material.

An expression for axial stress in the plastic due to thermal deformation may be derived from basic relations. If a strip of plastic and a strip of concrete are bonded together, the bonded strip will change in length with temperature. The individual strips, if unbonded, would also change in length with temperature, but by different amounts. Thus in a bonded strip each material will be deformed not only by temperature change but also by the other material. The load necessary to cause deformation of one material by the other is transmitted through the bond. The total deformation of either material equals the sum of its thermal deformation and its deformation caused by action of the other material.

$$\delta_b = \delta_t + \delta_p \quad (1)$$

where δ_b = total deformation of the bonded strips, in.

δ_t = unrestrained thermal deformation of one material, in.

δ_p = the deformation of one material caused by thermal elongation
(or contraction) of the other, in.

The total deformation, δ_b , of the plastic and concrete strips, when bonded, is the same, thus,

$$\delta_b = \delta_{t1} + \delta_{p1} = \delta_{t2} + \delta_{p2} \quad (1a)$$

where subscripts 1 and 2 refer to plastic and concrete respectively.

The expression for thermal deformation is:

$$\delta_t = \alpha L (T - T_o) \quad (2)$$

where α = thermal coefficient of expansion (or contraction) in/in ° C.

T = temperature °C

L = length, in.

The expression for load deformation (strain) of a material is:

$$\delta_p = \frac{PL}{AE} \quad (\text{Hooke's law}) \quad (3)$$

where P = load, lb.

A = cross-sectional area, sq. in.

E = modulus of elasticity, psi.

Substituting equation 2 and 3 in equation 1a yields:

$$\alpha_1 L(T - T_o) + \frac{PL}{A_1 E_1} = \alpha_2 L(T - T_o) + \frac{PL}{A_2 E_2} \quad (4)$$

In practice, a plastic strip will be applied to a concrete pavement, which will deform very little by action of the plastic. The last item of equation (4) is therefore negligible, and,

$$\alpha_1 L(T - T_o) + \frac{PL}{A_1 E_1} = \alpha_2 L(T - T_o) \quad (4a)$$

or

$$s = \frac{P}{A} = (\alpha_2 - \alpha_1) (T - T_o) E \quad (5)$$

where s = axial stress in plastic, psi.

subscript 1 has been dropped for E and A of the plastic, since these terms for concrete are no longer in the equation.

The sign of " s " in equation (5) follows the convention that tensile stresses are positive and compressive stresses negative.

The temperature T_o is one at which stress relief has been obtained by cold flow of plastic. This would normally be somewhat above the glass point of the plastic, at the upper limit of the temperature range in which the plastic has a fairly well defined modulus of elasticity. The thermoplastic materials, however, creep even at low temperatures; thus T_o might be close to the ambient temperature during a protracted period of cold weather. Because of this

characteristic, the most severe condition is anticipated to be a hard, sudden freeze, rather than a long cold spell.

Above a temperature of about 50° F, computed stresses have little meaning, since at such temperatures the plastics flow quickly enough to relieve them before they can build up. If T_0 has fallen to a low value during cold weather, a sudden warming could conceivably cause compressive stresses. Compressive stresses probably will not occur otherwise, since compression takes place under warming conditions.

A graphical illustration of stress variation with temperature is shown as Figure 2. Values have been estimated (from handbook references) for E and α . A plot based on experimental data might be somewhat different. Tension is indicated by a plus sign and compression by minus. No determination has yet been made of critical tensile stress, nor of the limiting level of compression stress; thus, these values are not shown.

Stresses in the bond between the thermoplastic and the concrete are under consideration, but no expression for them has been adopted.

IV. Formulation Studies

A. Proprietary Compositions

Proximate analysis of samples of "Permaline" (Permaline Corp.-Chicago), "Catatherm" (Cataphate Corporation-Toledo), and of California Division of Highways Specification "Experimental White Thermoplastic Traffic Tire Formula 1R353(1964)," as compounded in this laboratory was performed. The results are shown in Table I. It is interesting to note that the gloss bead content of the California Spec. is similar to "Permaline" while the resin content is similar to "Catatherm." Thus, the proximate composition of the California Spec. is seen to be somewhat intermediate between the proprietary materials.

B. Experimental Compositions

Compositional data on laboratory-prepared hot melt vehicles and compounds is shown in Table II. In this first series of experimental preparations, the California Specification was used as a known workable basis for a comparison of selected vehicle constituents. Of the vehicle compositions prepared, only #2 was omitted as unsuitable for pigmentation and testing.

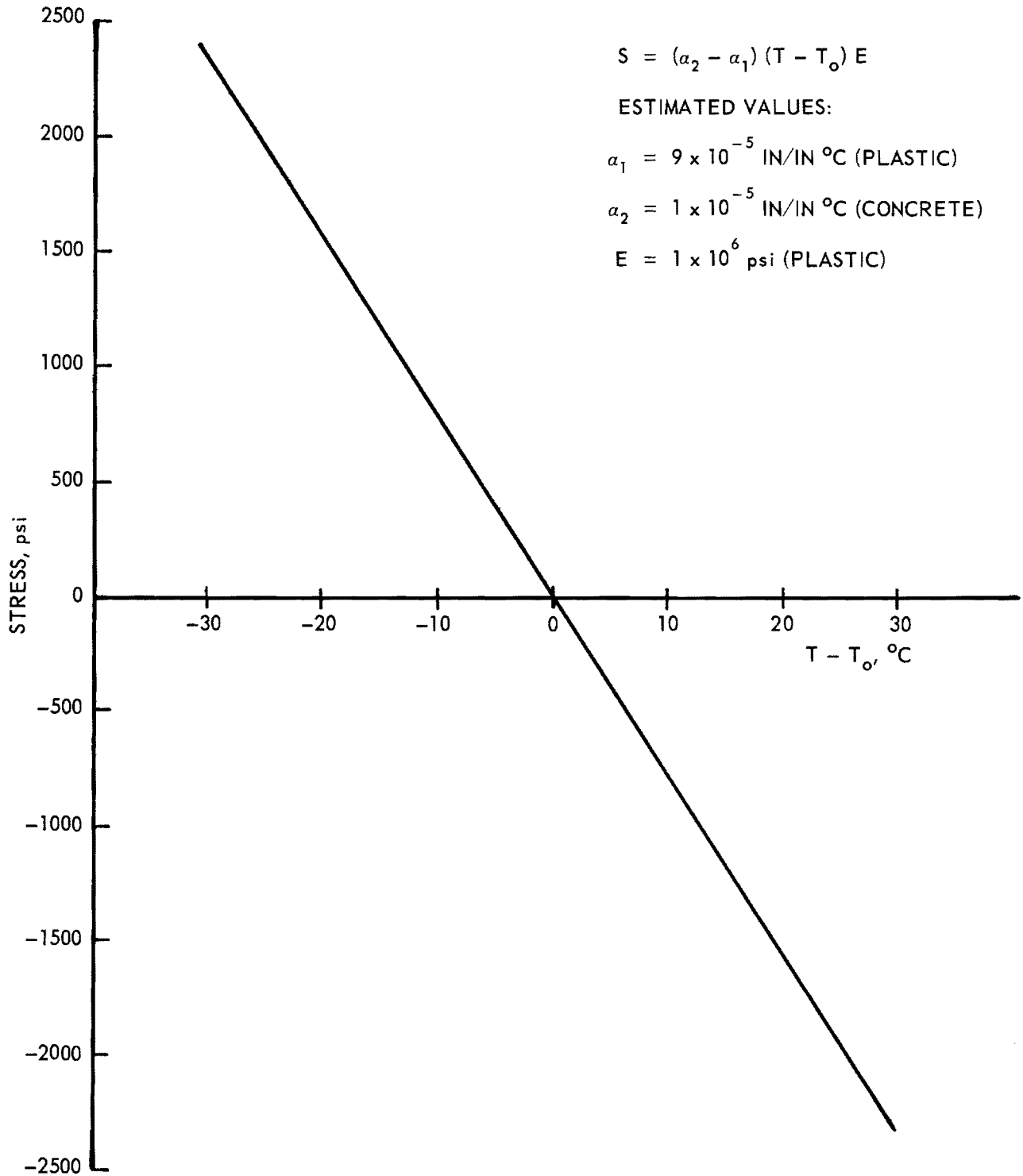


Figure 2. Computed Axial Stresses Resulting from Temperature Changes in Plastic Coating Bonded to Concrete.

TABLE I
HOT MELT PROXIMATE ANALYSIS

Material	Composition by Weight, Per cent		
	Permaline	Catatherm	Calif. Spec.
Glass beads	21	35	21
Pigment, excluding beads	64	40	54
Resins	<u>15</u>	<u>25</u>	<u>25</u>
Total Weight Per cent	100	100	100
Other Data			
Approximate bead size, mils	10	15	15
Phthalic acid, qualitative	present	present	absent

TABLE II
EXPERIMENTAL HOT MELT VEHICLES AND COMPOUNDS

Formulation No.	Composition in Grams					
	1	2 [*]	3	4	5	6
<u>Vehicle</u> ^{**}	(Calif. Sp.)					
Stabilite No. 10 (1)	98	-	-	-	-	160
Pliolite AC L (2)	90	90	90	90	90	-
Abitol (1)	58	58	58	58	58	-
Piccopale H-2 (3)	-	98	-	-	-	-
Piccotex 120 (3)	-	-	98	-	-	-
Piccotex 100 (3)	-	-	-	98	-	-
Ethocel (1)	-	-	-	-	-	50
(5cps, Ethoxy 48.8%)						
Stikvel W (5)	-	-	-	-	98	-
Castor Oil #3 (4)	-	-	-	-	-	37.5
Tricrysyl Phosphate	-	-	-	-	-	2.5
<u>Pigments</u>						
Glass Beads	208	-	208	208	208	208
Rutile TiO ₂	130	-	130	130	130	130
CaCO ₃ , R040	416	-	416	416	416	416

* #2 vehicle was quite cloudy and was omitted from further tests.

** Sources:

- (1) Hercules Powder Company
- (2) Goodyear, Chemical Division
- (3) Pennsylvania Industrial Chemical Corp.
- (4) Baker Castor Oil Company
- (5) Velsicol Chemical Corporation

All of the tabulated vehicle compositions were first prepared in larger quantity without pigmentation by melting and mixing the vehicle ingredients in a metal container on a hot plate. The melt was then allowed to cool and solidify in a metal pan, and any indications of incompatibility in the solid mass was noted. A weighed quantity of the solid composite vehicle was then remelted and combined with the pigments by hand stirring with a spatula. Only minor variations in dispersability were noted except for Formulation No. 6 which exhibited a tendency for lumpiness. Prepared composite vehicles and pigmented formulations were retained for physical property tests.

C. Physical Property Tests

Viscosity-temperature relations have been determined for proprietary formulations, for experimental formulations, and for experimental composite vehicles. In each case: (1) a specimen was melted and heated to 460° F on the hot plate, (2) Brookfield Viscometer spindle #6 operating at 20 rpm was introduced into the melt, (3) the hot plate was removed, and (4) temperatures and viscosities were recorded at intervals as the melt slowly cooled to about 300° F.

It was found that the experimental data could be correlated to a good approximation as straight lines on an Arrhenius plot (log viscosity versus reciprocal absolute temperature). The data is presented in this manner in Figure 3. Some departures from straight line correlations could be anticipated on theoretical grounds and are indicated by the data, but these rheological-structural details are not of immediate engineering interest. The various materials (excepting #5 which has not been run) are identified by name or formulation number on the plot. The clear (unpigmented) vehicles are identified by the formulation number with a suffix "V", and are seen to be grouped in a lower viscosity range of the plot. The viscosity-temperature characteristics of the vehicles based on Pliolite ACL were very similar. Formulation No. 6V based on ethyl cellulose exhibited lower viscosity and steeper slope. The proprietary and experimental pigmented compositions are grouped in a higher viscosity range of the plot. The California Specification (#1) is fairly similar to both proprietary compositions. In general, the pigmented experimental formulations tend to exhibit viscosity characteristics which parallel their respective vehicles.

The viscosity variations observed do not appear to be large enough to produce noticeable differences in application characteristics. Satisfactory test applications to wear test discs were achieved with all materials.

Testing work has been started on accelerated wear, flexural creep, and falling ball impact resistance, but these tests have not progressed sufficiently to report at this time.

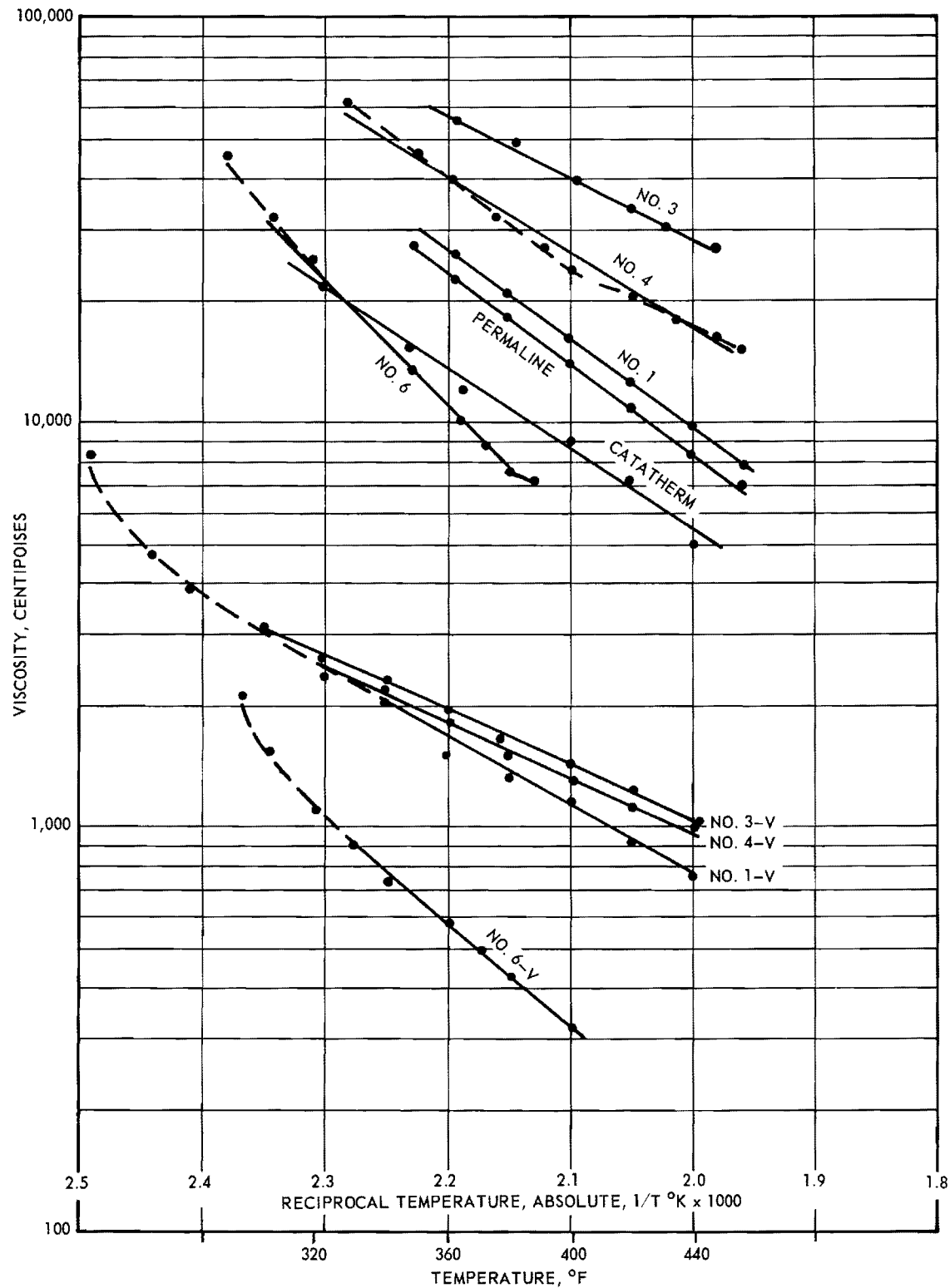


Figure 3. Temperature-Viscosity Relations.

V. Future Work

Immediate emphasis continues to be directed towards achieving initial field applications as soon as possible. Subject to acquisition and check out of minor equipment items, this objective should be accomplished well within the next quarterly period. The hot melt compositions already compounded with certain selected variations to attain a greater range of performance potential, should be entirely adequate for this initial field application.

Current laboratory work is intended to emphasize development of appropriate testing methods. Increasing attention will be directed toward systematic formulation studies as confidence is gained in interpretation of laboratory test methods.

Respectfully submitted:

W. R. Tooke, Jr.
Project Director

Approved:

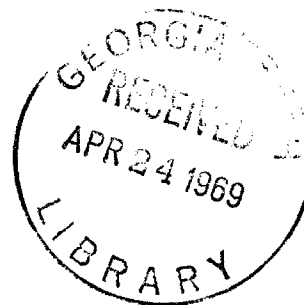
W. H. Burrows, Head
Industrial Products Branch

GEORGIA INSTITUTE OF TECHNOLOGY

ENGINEERING EXPERIMENT STATION

ATLANTA, GEORGIA 30332

July 16, 1965



Division of Highway Planning
State Highway Department of Georgia
Atlanta, Georgia

Attention: Mr. H. H. Huckeba
State Highway Planning Engineer

Subject: Quarterly Progress Report No. 2, Project A-802
"Hot Melt Traffic Marking Materials"
Contract No. 6401
Covering the Period from March 1 to June 30, 1965

Gentlemen:

Project activities for this quarterly report period have been conducted in the following areas:

- I. Formulation Studies
 - A. Experimental Compositions
 - B. Physical Property Data
- II. Equipment Development
- III. Highway Test Applications
- IV. Future Planning

Additional details of these activities follow.

I. Formulation Studies

A. Experimental Compositions

Hot Melts. Formulation work during the current period was oriented specifically toward producing a small set of candidate hot melt compositions suitable for highway testing. While it is mandatory that all such compositions be capable of application under standard conditions, in this series it was desired also that physical properties exhibit sufficient range to provide some conception of their relation to highway performance. After culling a larger set of preparations to reduce the scope of the field study to manageable size, six formulations were chosen for the field program. Formulation details are given in Table I.

The "California Spec.," No. 15, was included because of accumulated experimental and usage background. Variations in the resin proportions of this composition, Nos. 16 and 19, were included to

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TABLE I
HOT MELTS FOR HIGHWAY TESTS

Formulation No.	Composition in Per Cent by Weight					
	$\frac{1}{2}$ S/A 16	Cal. Spec. 15	2 S/A 19	Sp. Hydrocarbon 21	Et. Cel. 24	Alkyd 22
Vehicle*						
Abitol (1)	5.51	5.79	5.50	-	5.65	-
Plidlite A.C. (milled) (2)	4.30	8.99	17.27	8.99	-	-
Stabilite # 10 (1)	13.58	9.79	0.76	-	9.81	9.82
Stikvel W (3)	-	-	-	15.58	-	-
VBR 5010 (4)	-	-	-	-	-	9.82
Pentalyn K (1)	-	-	-	-	-	4.93
Ethyl Cellulose W-4 (1)	-	-	-	-	9.08	-
Pentylphenol (5)	-	-	-	-	0.10	-
Fillers						
Glass beads	21.14	20.81	21.10	20.81	20.79	20.81
TiO ₂ -R-610	13.19	12.99	13.17	12.99	12.97	12.99
RO-40 (CaCO ₃)	42.26	41.62	42.20	41.62	41.58	41.62

- * Materials Sources
- (1) Hercules Powder Co.
 - (2) Goodyear Tire and Rubber Co.
 - (3) Velsicol Chemical Corp.
 - (4) Union Bag Camp Paper Corp., Nelio Chemical Div.
 - (5) Eastman Organic Chemicals

introduce deliberate physical property variations. The other three formulations were chosen in part to attain further variety in physicals, but also to demonstrate the capabilities of other resin systems. Formulation No. 21 in place of the rosin-derived resins in the California Specification embodies a special hydrocarbon resin that appears to have outstanding adherence characteristics. Ethyl cellulose was the basic polymer of Formulation No. 24. This has been a "classical" material for hot melt adhesives and related applications for many years, and one producer of this resin has done some experimental work on traffic marking applications. Formulation No. 22 is based on a non-drying alkyd resin. A material of this general type is cited in the patent literature, and it appears to be the basis of proprietary formulations.

It will be observed that this study involves variations only in the resin vehicle component of the total formulation. This is not to imply that the pigment loading is to be ignored in future work. We have noted, however, that hot melts do not lend themselves to the wide range of pigment loadings that are feasible with paints containing solvents. In general, the maximum loading attainable consistent with satisfactory application characteristics appears to be indicated for optimum performance. At lower loadings increased shrinkage and poorer dimensional stability are usually observed. Since guiding principles are available for aggregate proportioning, this subject is to be investigated in more detail in the laboratory.

Primers. Two types of primers were of immediate interest for the highway studies--a nitrile rubber/phenolic material and a catalyzed epoxy material. The former was available from the Goodyear Chemical Division under the designation Pliogrip AD 574. This product conforms with California Specification 63-F-40.

Catalyzed epoxy primers are produced by the vendors of hot melt marking compositions, and formulation details are not available from these sources. A sample was obtained from the traffic department of the City of Atlanta and studied for its properties. On the basis of the information obtained from this examination, we were able to assemble a formulation of essentially identical properties having the following composition:

<u>Epoxy Primer</u> *		
Component A (Epoxy)		50% by weight
Epon 828 (Shell Chem. Co.)	50%	
Acetone	50%	
Component B (Polyamide)		50% by weight
Curing Agent V-25 (Shell Chem. Co.)	50%	
Acetone	50%	

* Mix Components A and B thoroughly just prior to application--pot life about 6 hours. Hot melt may be overlaid within 5 minutes.

B. Physical Property Data

Systematic work on physical property characterization was subordinated to the urgent activity on field formulations during the current period. An impact tester of special design has been ordered (EES Capital Funds) for the Industrial Products Branch, and we anticipate extensive use of this device to study impact properties. In addition, we have acquired a gradient bar melting point apparatus to rapidly characterize resin softening points. Another recent acquisition is a freezer chest which is to be used for low temperature testing.

Some experimental work has been done on flexural creep properties of hot melts. Plastic specimens for this test are cast in a mold to dimensions of $\frac{1}{4}$ " x $1\frac{1}{4}$ " x 8". The specimens are tested in a simple jig involving center point constant loading on a span of 4 inches. Test time and deflections are recorded and plotted as shown in Figure 1. For specimens tested, it has appeared that relative creep properties closely parallel hot melt viscosities. This is certainly not surprising, but we cannot be certain that the relationship will be maintained when we examine creep at lower temperatures. Attention is called to the line on the plot identified as Permaline "CA"*. This material was a sample obtained from the City of Atlanta, and its temperature-viscosity characteristics were shown in Figure 3 of Quarterly Progress Report No. 1 (3/10/65). Note that Permaline 11J63CS5A** subsequently purchased from the manufacturer for use in the highway tests, exhibits drastically higher creep characteristics. Viscosity-temperature curves have not been run on this material, but it is assumed that corresponding differences will be observed.

II. Equipment Development

A. Field Applicator

The field applicator was modified to provide quick change adjustments of the extruder gate to yield clearances of $3/16$ " or $3/32$ ". Numerous trial applications were run and further adjustments and minor modifications were made prior to the highway test application work.

B. Premelter

Construction of the premelter was completed generally along the lines described in the previous Quarterly Report No. 1, page 3, (3/10/65). It was necessary to scale the container size upward from 1 gallon to 5 gallons to accommodate the hot melt quantities required for the highway test applications. The unit was thoroughly tested prior to the field work and was found capable of heating a batch of 3 gallon size to application temperature in about one hour without excessive overheating. Batches could also be maintained at application temperatures for several hours with a minimum of attention.

* Sample obtained from City of Atlanta.

** Sample purchased from Permaline.

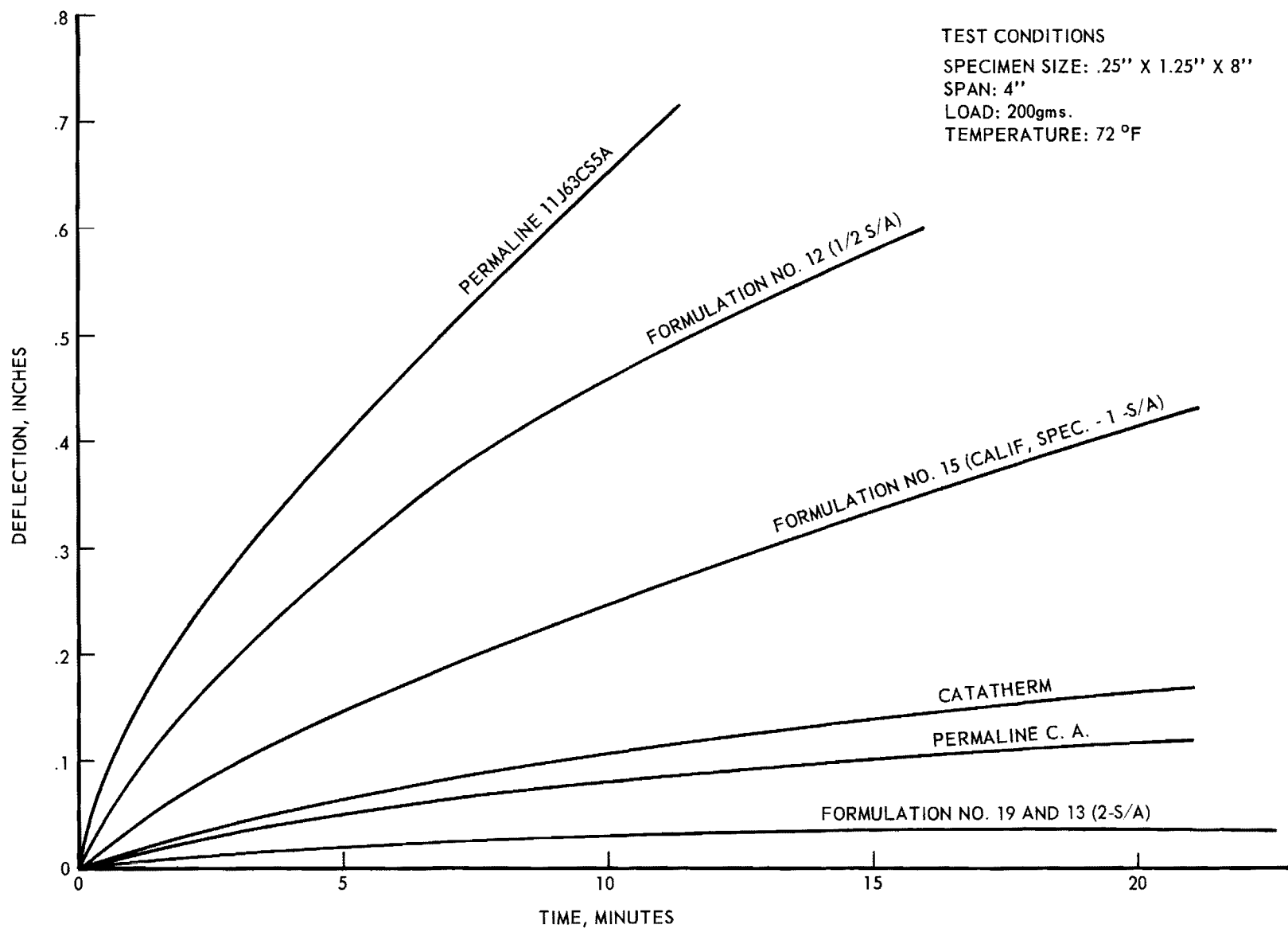


Figure 1. Flexural Creep Tests.

C. Compounding Equipment

A drill press was fitted with a variable speed drive unit and a heavy duty paddle impeller was attached to the chuck. One gallon stainless steel beakers were utilized for mixing containers, and heating was provided by a hot plate. This compounding equipment was found to provide very satisfactory resin melting and filler blending.

III. Highway Test Applications

On June 1-3, 1965, test applications were made on Interstate 85 at Lenox Road Bridge. All formulations were successfully applied; however, some difficulty was experienced with high viscosity characteristics of Formulation No. 19 (double concentration of Pliolite AC). A total of 68 hot melt stripes and 12 control paint stripes were placed. Photographs of the application work are shown in Figure 2.

A detailed plan of this experiment is presented in Table II. In general, priming has been considered unnecessary on asphalt paving; however, priming was included on four of the lines on asphalt (Lines 356, 357, 359, 360) to evaluate a possible effect.

The column headed "Nominal Application Thickness" refers to the gate clearance of the applicator while the "Measured Thickness" designates thickness measurements made on the stripes in place using a surface gage mounted on a bridging frame. Although the roughness of the concrete affects the precision of the measurements, the data indicate rather positively that significant thickness differences must have been caused by differences in the flow characteristics of the several materials. It was not really anticipated that precise thickness control could be achieved. Subsequent performance interpretations may be rendered more precise by "normalizing" the data to a constant stripe thickness if such procedures appear desirable.

IV. Future Planning

Emphasis is currently being directed to the completion of physical property test methods and to the relationships of formulation variables to physical properties. It is anticipated that this work will represent the major project activity for the next several months.

Respectfully submitted,

W. R. Tooke, Jr. //
Project Director

Approved:

W. H. Burrows, Head
Industrial Products Branch



Figure 2. Photographs of Highway Application Work.

TABLE II
HOT MELT HIGHWAY APPLICATION DATA (6/1/65)

Line No.	Formulation Number	Substrate C - Concrete A - Asphalt	Priming		Nominal Application Thickness, inches	Measured Thickness, inches
			O - None	R - Rubber E - Epoxy		
301	Permaline	C	O		.094	.105
302			R			
303			E			
304			O		.188	.130
305	Catatherm	C	R			
306			E			
307			O		.084	.068
308			R			
309			E			
310			O		.188	.118
311			R			
312			E			
313	H. M. Formula # 16 ($\frac{1}{2}$ S/A)	C	O		.094	.103
314			R			
315			E			
316			O		.188	.133
317			R			
318			E			
319			O		.094	.088
320			R			
321	# 15 (Calif. Spec.)		E			
322			O		.188	.118
323			R			
324			E			
325	H. M. Formula # 19 (2 x S/A)	C	O		.094	.113
326			R			
327			E			
328			O		.188	.124
329			R			
330			E			
331			O		.094	.088
332			R			
333	H. M. Formula # 21 (H'carbon Resin)	C	E			
334			O		.188	.128
335			R			
336			E			
337	H. M. Formula # 24 (Eth. Cell.)	C	O		.094	.073
338			R			
339			E			
340			O		.188	.118

(continued)

TABLE II (concluded)

HOT MELT HIGHWAY APPLICATION DATA (6/1/65)

Line No.	Formulation Number	Substrate C - Concrete A - Asphalt	Priming		Nominal Application Thickness, inches	Measured Thickness, inches
			O - None	R - Rubber E - Epoxy		
341	# 24 (Eth. cell.)			R		
342				E		
343	H. M. Formula	C		O	.094	.123
344	# 22 (alkyd)			R		
345				E		
346				O	.188	.113
347				R		
348				E		
349	Paint No. 95	C		O	.010	
350	(Alkyd)			O	.015	
351				O	.020	
352	Paint No. 96	C		O	.010	
353	(Parlon-Alkyd)			O	.015	
354				O	.020	
355	Permaline	A		O	.094	.073
356				R		
357				E		
358				O	.188	.093
359				R		
360				E		
361	Catatherm	A		O	.094	.063
362				O	.188	.173
363	H. M. Formula	A		O	.094	.083
364	# 16 ($\frac{1}{2}$ S/A)			O	.188	.118
365	H. M. Formula	A		O	.094	.068
366	# 15 (Calif. Spec.)			O	.188	.113
367	H. M. Formula	A		O	.094	.093
368	# 19 (2 S/A)			O	.188	.118
369	H. M. Formula	A		O	.094	.138
370	# 21 (Stikvel)			O	.188	.133
371	H. M. Formula	A		O	.094	.073
372	# 24 (Eth. Cell.)			O	.188	.113
373	H. M. Formula	A		O	.094	.073
374	# 22 (Alkyd)			O	.188	.128
375	Paint No. 95	A		O	.010	
376	(Alkyd)			O	.015	
377				O	.020	
378	Paint No. 96	A		O	.010	
379	(Parlon-Alkyd)			O	.015	
380				O	.020	

GEORGIA INSTITUTE OF TECHNOLOGY

ENGINEERING EXPERIMENT STATION

ATLANTA, GEORGIA 30332

October 11, 1965



Division of Highway Planning
State Highway Department of Georgia
Atlanta, Georgia

Attention: Mr. Emory C. Parrish
State Highway Planning Engineer

Subject: Quarterly Progress Letter 3, Project A-802
"Hot Melt Traffic Marking Materials"
Contract No. 6401
Covering the Period from 1 July to 30 September 1965

Gentlemen:

Project activities during this quarterly report period were conducted primarily in the following areas:

- I. Development of Physical Test Apparatus and Procedures
 - A. Tensile and Shear Bond Strengths
 - B. Static Stress-Strain Studies
 - C. Thermal Coefficient of Expansion
- II. Formulation Studies - Filler Packing Density
 - A. Particle Packing Theory
 - B. Packing Density Measurement
 - C. Densities of Composite Fillers
 - D. Compounding Effects
- III. Inspection of Highway Applications

Details of these activities follow:

I. Physical Tests

Major project activity has been directed to the development of suitable physical testing procedures. The work has included development of specimen molding techniques, testing assemblies, and some preliminary test findings.

A. Tensile and Shear Bond Strengths

The molding procedure for test specimens is illustrated by Figures 1 and 2. Testing assemblies for static tensile and shear bonds are shown in Figures 3 and 4, respectively. The hot melt is molded to dimensions of 2 x 2 inches in cross-section and 0.25 inches thick between a

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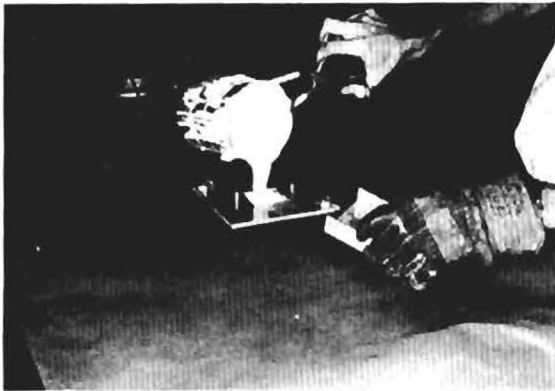


Figure 1. Pouring hot melt into molding form for bond tests.

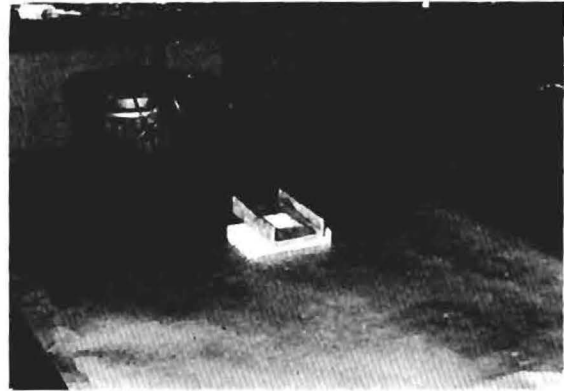


Figure 2. Completed bond tests specimen.

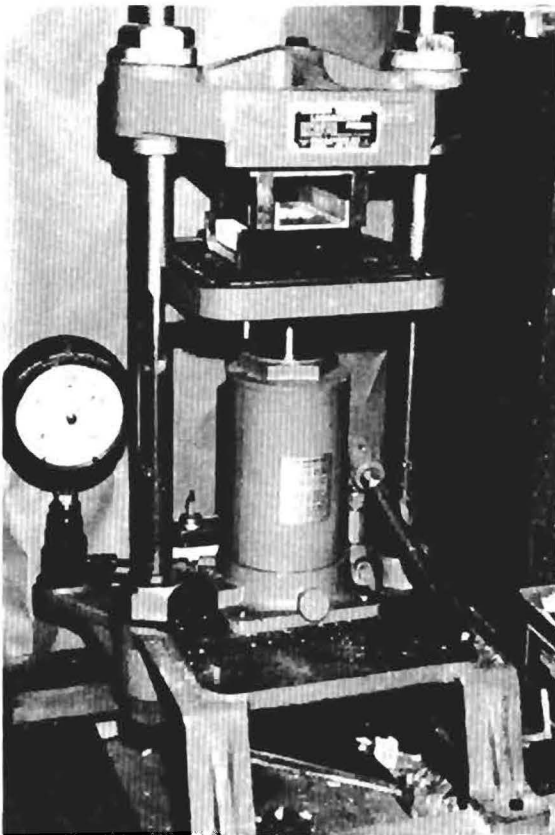


Figure 3. Tensile bond test assembly.

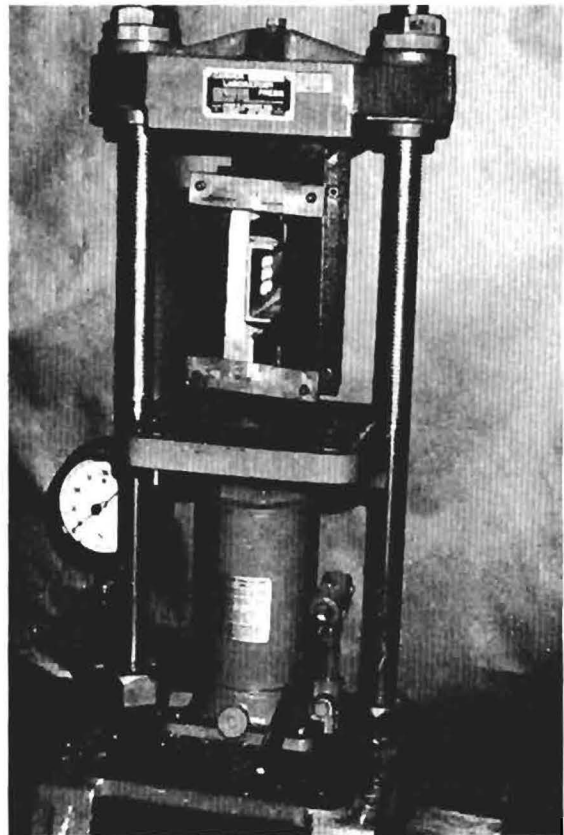


Figure 4. Shear bond test assembly.

concrete block and a perforated aluminum channel. The use of the channel was indicated as a means of reducing the mass for subsequent impact testing, and of confining a given test to a single hot melt-concrete bonding interface. Moreover, an anticipated large consumption of concrete blocks was thereby reduced by half.

Some very interesting results have been obtained from preliminary tests. We have found that the bond of Pliogrip primed concrete increases substantially during a three day period following molding. Epoxy primed concrete exhibits essentially no bond for several hours after molding, but the bond builds up to high levels during a three day period after molding. Erratic bonding results on bare concrete suggested another type of test. Concrete blocks were immersed in water overnight, removed, allowed to stand in the laboratory for about two hours until the surface was dry, and then bonded into test assemblies. The effective tensile bond of these assemblies was zero--some fell apart upon mold stripping. By comparison, the average bond strength with dry blocks was 55 psi.

Additional preliminary tests are in progress to establish standardized test conditions, and, thereafter, all hot melt compositions of interest will be subjected to these tests.

B. Static Stress-Strain Studies

Initial exploratory work to measure static tensile stress-strain of hot melts was pursued in accordance with ASTM D-638-61T Tensile Properties of Plastics. A series of tests performed on equipment owned by Instron Engineering Corporation indicated that reliable tensile stress-strain data cannot be obtained without extensometer equipment which is not presently available on our machine. Therefore, we decided to utilize a flexural test which obviates the gage length problem of the tensile test and provides reliable stress-strain data without extensometers. This flexural test is conducted in accordance with ASTM D-790 Flexural Properties of Plastics. Some preliminary test results indicate that the flexural stress-strain responses of hot melts are extremely rate-sensitive. For example, at a deflection rate of 1 in./min. plastic flow occurred with no rupture, and a maximum load of only 72 psi (maximum fiber stress = 691 psi) was observed. In contrast with this, at a deflection rate of 12 in./min. brittle rupture occurred at 104 psi (maximum fiber stress = 1000 psi) with very small deflection. It is anticipated that the 12 in./min. deflection rate may be adopted as standard for this test so that brittle rupture will be observed. Thus the complete testing program of stress-strain characterization will include flexural data at three distinct displacement rates as follows:

Creep flexure	-	extremely slow	10^{-2} in./min.
Static flexure	-	moderate rate	10^1 in./min.
Impact flexure	-	very fast	10^4 in./min.

These rates should encompass all conditions of practical interest.

C. Thermal Coefficient of Expansion

A suitable device was constructed to measure coefficient of linear thermal expansion in accordance with ASTM D-696-44. Routine measurements have been initiated.

II. Formulation Studies - Filler Packing Density

A. Particle Packing Theory

By computing or determining optimum distributions of particle sizes, the void volume of granular material may be very substantially reduced, with a corresponding increase in apparent density. The void volume parameter is utilized effectively in some very successful schemes of concrete mix design, and it finds use in paint technology in the form of the critical pigment volume concentration (CPVC). In hot melts, the use of void volume ideas offers the potential for significantly reducing the cost of compositions, while improving application and performance characteristics. The particle packing theories are discussed in considerable detail by Dalla Valle*. The practical objective is to attain by computation or experiment a distribution of particle sizes such that increasingly finer particles can most completely occupy all void spaces between coarser particles. Computational approaches to the problem are based on assumptions of spherical particles and, therefore, can provide only general guidance for practical work. In practice, systematic experimental determinations of packing densities are always required.

B. Packing Density Measurement

In hot melt compositions of current interest the filler constituent has the following composition:

	<u>Wt. %</u>
Titanium Dioxide	17.2
Ground Marble (R0-40)	55.2
Glass Beads (coarse)	27.6

The titanium dioxide in this filler is of such fine particle size (0.5μ) that it may be regarded appropriately as a part of the resinous matrix, and thereby ignored as a filler constituent. Attention may thus be concentrated on the mixture of ground marble and glass beads. These relatively coarse materials are also more amenable to the measurement method as outlined below:

Measurement Method

Selected quantities of the filler components are individually weighed and combined to yield about 400 grams of composite filler. The composite filler is violently agitated on a paint shaker in a one quart can for five minutes, and then poured into a 500 cc graduated cylinder. The cylinder bottom is then tapped (bounced) uniformly for 25 strokes on a rubber mat to attain maximum consolidation. Final volume is observed and apparent density (weight/volume) computed.

C. Densities of Composite Fillers

The first study of packing densities was undertaken on the premise that higher density might be attained by incorporating some

* J. M. Dalla Valle, Micromeritics, Chapter 6, Pitman Publishing Corp., New York (1948).

fine glass beads in the mixture. The apparent density response surface for the system Ground Marble (RO-40)-Coarse Beads-Fine Beads is shown in Figure 5 on a ternary plot of percentage composition by weight with the apparent density parameter presented in the form of contour lines. In this investigation it was discovered that at the maximum density point, only 25% of ground marble is combined with a total of 75% of coarse and fine beads. This was believed to be an excessive bead concentration. Furthermore, the use of fine rather than coarse beads to obtain maximum retro-reflection is indicated in the work of Pocock and Rhodes*. Therefore, the use of a coarse ground marble to replace the coarse beads was proposed.

A suitable coarse ground marble designated as Ground Marble, XO was obtained, and another ternary study was completed on the system Ground Marble, XO-Ground Marble, RO-40-Fine Beads as shown in Figure 6. This time maximum density was obtained in a compositional area of about 20% of fine glass beads, but up to 40% might be used without serious loss of packing efficiency.

The general significance of these findings is that optimum packing densities may be readily determined and should provide very helpful guidance in selecting filler compositions.

D. Compounding Effects

At present, this work has been barely initiated, but the findings appear most significant. The filler composition represented by the maximum density point in Figure 5 was substituted in a control (California Specification - Hot Melt No. 1) on a weight replacement basis. The temperature viscosity curve of the resulting Hot Melt No. 25 was then determined. The result is shown in Figure 7 as compared with the control. Note the very significantly lower viscosity of the new composition. This appears to be a clear demonstration of the reduction of the "binder demand" of a hot melt formulation by the adjustment of filler size distribution to obtain minimum voids.

III. Inspection of Highway Applications

On September 8, 1965, the highway cross-stripe application tests placed on June 1, 1965, were inspected for integrity and night visibility. Wear and weathering effects were almost negligible with respect to film integrity. A random occurrence of patterns of circular cracks approximately 2 inches in diameter in several of the stripes has not been satisfactorily explained. One theory, which will be checked on the next inspection, is that these cracks surround spots of tar on the concrete where adhesion was poor and stresses accumulated. Night visibility

* B. W. Pocock and C. C. Rhodes, "Principles of Glass-Bead Reflectorization," Highway Research Board Bulletin No. 57, p. 39, 1952.

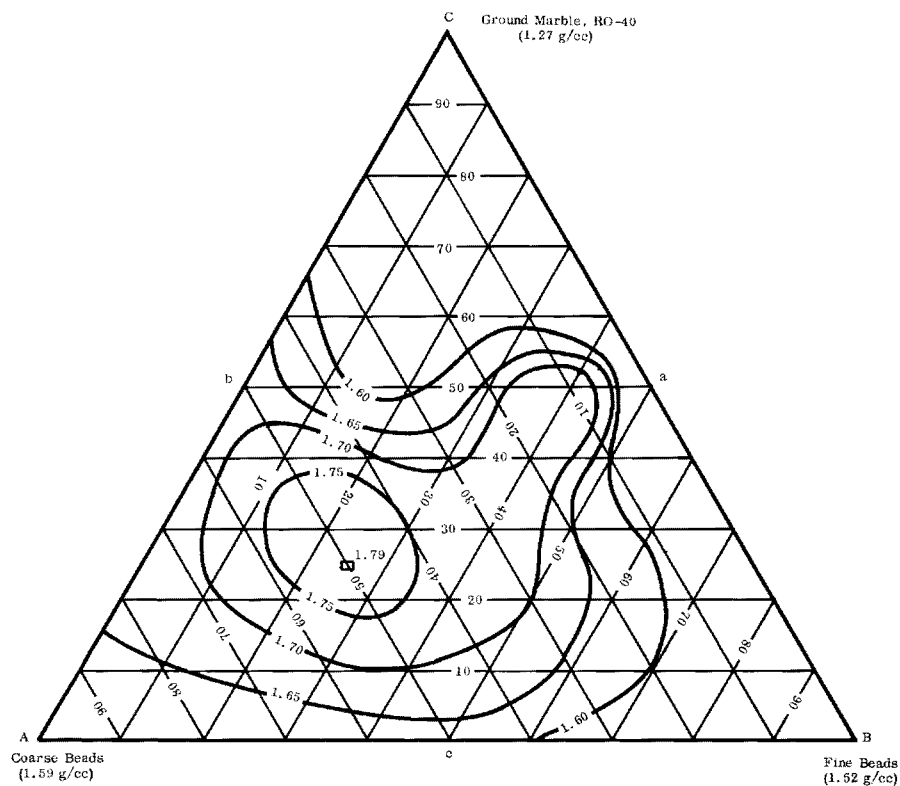


Figure 5. Apparent Density of the System Ground Marble, RO-40 - Coarse Beads - Fine Beads

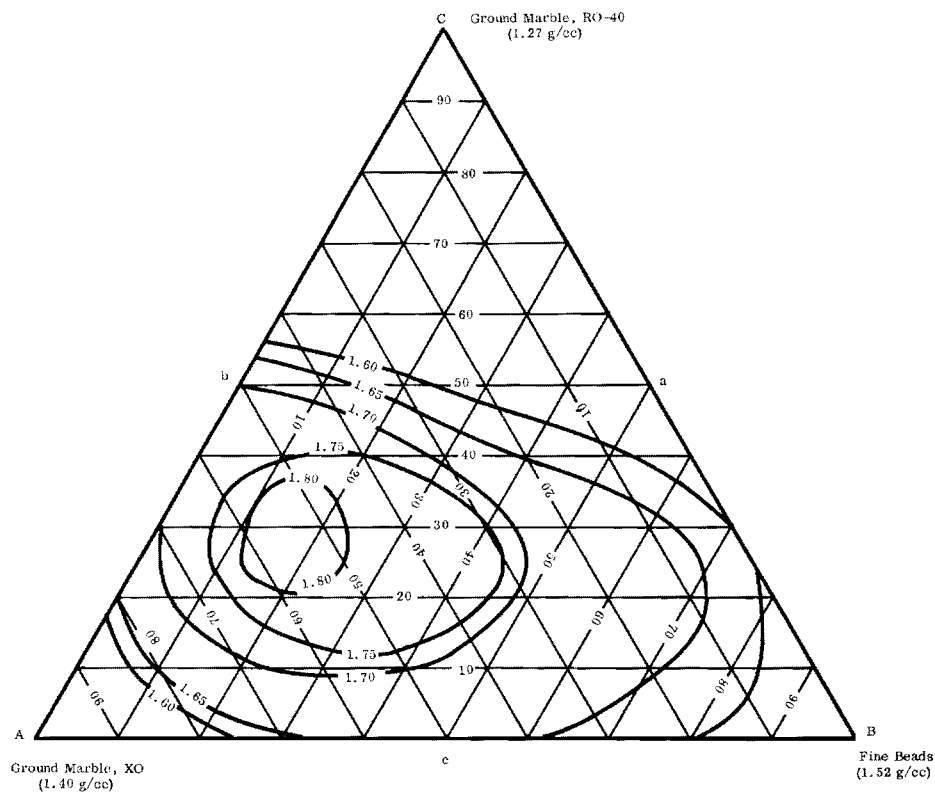


Figure 6. Apparent Density of the System Ground Marble, RO-40 - Ground Marble, XO - Fine Beads

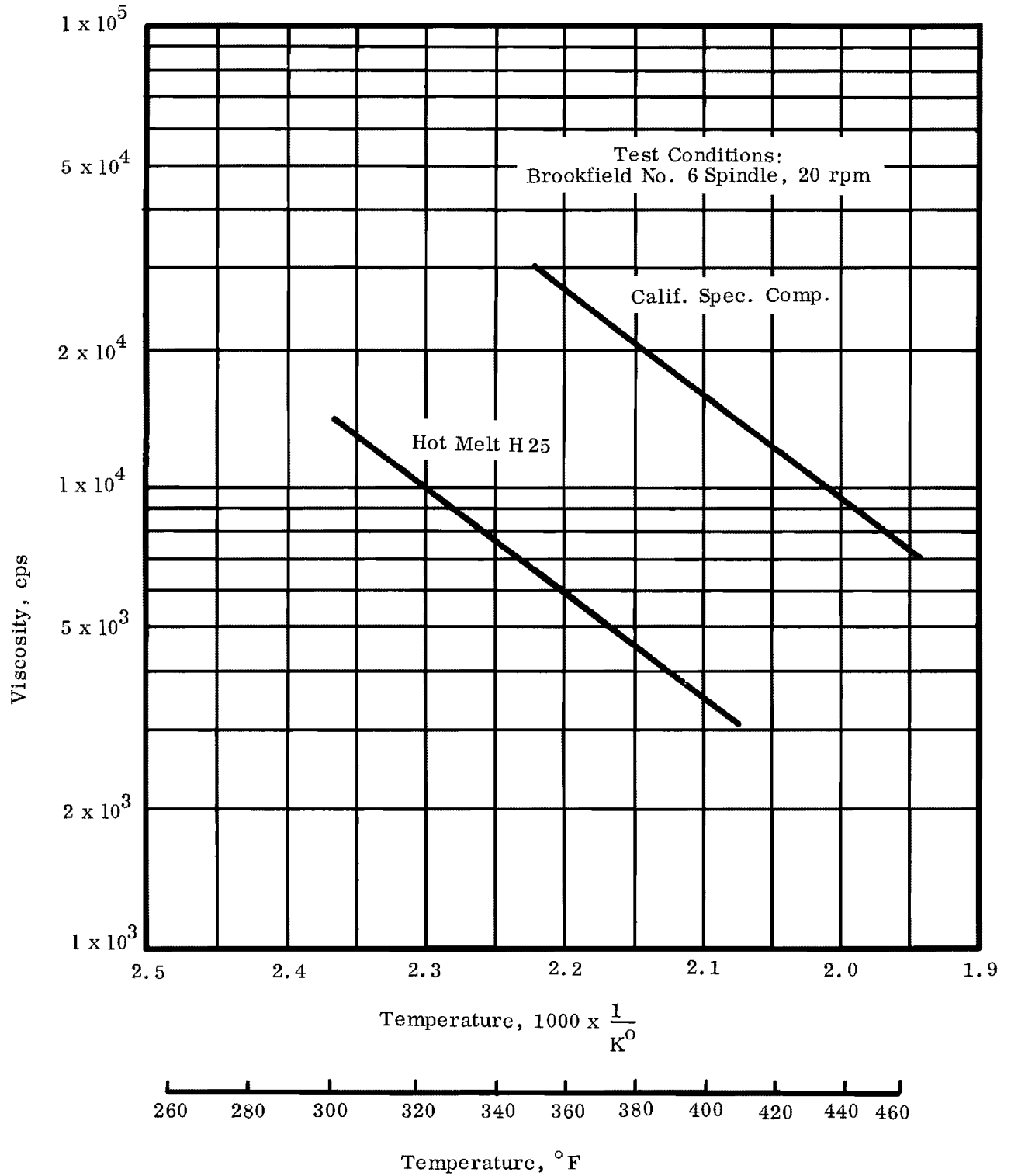


Figure 7. Effect of Filler Substitutions on Viscosity

exhibited a general deterioration over the three months period. Differences among the formulations were indicated, but were not considered to be conclusive. All lines were above the level of 10 which is considered the minimum of useful reflectance.

Respectfully submitted,

W. R. Tooke, ~~Dr.~~
Project Director

Approved:

W. H. Burrows, Head
Industrial Products Branch

GEORGIA INSTITUTE OF TECHNOLOGY

ENGINEERING EXPERIMENT STATION

ATLANTA, GEORGIA 30332

January 10, 1966

Division of Highway Planning
State Highway Department of Georgia
Atlanta, Georgia

Attention: Mr. Emory C. Parrish
State Highway Planning Engineer

Subject: Quarterly Progress Letter No. 4, Project A-802
"Hot Melt Traffic Marking Materials"
Contract No. 6401
Covering the Period from 1 October to 31 December 1965

Gentlemen:

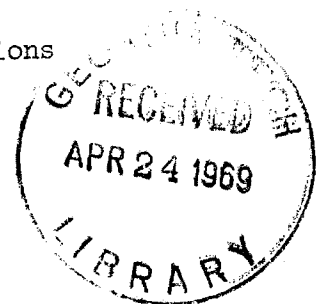
Project activities during this quarterly report period were conducted primarily in the following areas:

- I. Formulation Studies
 - A. Comparative Properties of Highway Test Compositions
 - B. Titanium Dioxide Pigmentation
 - C. Systematic Studies of Vehicle Additives
- II. Test Development Work
 - A. Hot Melt Consistency
 - B. Impact Tests
 - C. Flexural Elastic Modulus and Creep
- III. Inspection of Highway Applications

Details on these activities follow.

I. Formulation Studies

During the previous quarter (Quarterly Report No. 3), we developed the "void volume" approach to the selection and proportioning of fillers in hot melts. Subsequently, we have completed sufficient laboratory testing to permit comparisons among the various highway test formulations, and to evaluate the new approach to filler proportioning. The quantitative requirements for the opacifying pigment (TiO_2) have also been established. Attention was then directed to the hot melt vehicle composition. The results of the systematic vehicle studies were being accumulated concurrently with preparation of this report, therefore the findings presented have not been analyzed in detail.



A. Comparative Properties of Highway Test Compositions

The formulations discussed in this section were given in detail in Quarterly Progress Report No. 2 (7/16/65). The original retained samples of the highway tests were insufficient for the complete series of laboratory tests, therefore, larger quantities of three selected formulations were prepared. These are identified by new formulation numbers followed by the original numbers in parentheses.

Test data are shown in Table I for the three highway test compositions together with proprietary compositions. Information on the test methods is given in Section II of this report.

The data suggest that the properties of the experimental materials, in general, are not greatly different from the proprietary compositions. All formulations were found to exhibit low bond strengths at 0°F. Hot melt # 27 (24) based on ethylcellulose was observed to have outstanding impact resistance.

B. Titanium Dioxide Pigmentation

Levels of TiO_2 pigmentation between 0% and the 13% utilized in the Calif. Spec. (#26) were explored experimentally. Opacity and whiteness was judged to be adequate at a TiO_2 level of 3.5%. Vehicle color has been observed to affect whiteness significantly; therefore, we have concluded that TiO_2 requirements should be based on appearance properties rather than an arbitrary compositional percentage. This can permit more economical utilization of this relatively costly ingredient.

C. Systematic Studies of Vehicle Additives

This work was begun in November prior to the completion of the physical tests on the "Highway Test Compositions" reported above. Accordingly, the selection of a "basis formulation" was made primarily from considerations of economy and compatibility as conditioned by highway performance observations and some laboratory tests. These considerations clearly favored an alkyd-type formulation, and it would appear that subsequent work at least partially supports the validity of this choice.

The purpose of this study was to evaluate in the alkyd-type formulation the individual effects on physical properties of various vehicle additives. Each additive was incorporated by substitution at a level of 10% by weight of the original composite vehicle. This substitution treatment included the original components of the formulation as well as other resins. Formulation details are given in Table II.

The results of this study are shown in Table III. Details of the special test methods are discussed in the next section. Two "master batches" of the basis formulation were prepared. The first, H.M. # 39, was used to prepare the variations represented by H.M. # 36, 37, 38, and 40. The second, H.M. # 42, was used to prepare H.M. # 41, 43, 44, 45, 46, 47, 48, 49, 50, 51, and 52. The poor agreement of test results of

TABLE I
PHYSICAL TESTS OF HIGHWAY TEST COMPOSITIONS

Hot Melt No.	Description	Density (wt./gal.)	Consistency @ Slope		Elastic Modulus (psi x 10 ⁵)	Flexural Creep Properties		Static Shear Bond Strength			Flexural Impact Tests			
			350°F (cps)	@ 350°F (cps/°F)		Initial Elastic Deformation (in./in. x 10 ⁻³)	Creep Rate (in./in./min. x 10 ⁻³)	@ 77°F (psi)	@ 0°F (psi)	Change (%)	Max. Fiber Stress @ 77°F (psi)	Absorbed Energy @ 77°F (in.lb./ in. ²)	Max. Fiber Stress @ 0°F (psi)	Absorbed Energy @ 0°F (in.lb./ in. ²)
Permaline		17.3	7,500	390	3.8	1.5	5	-	-		1630	1.65	2100	2.7
Catatherm		18.1	22,400	330	2.3	2.0	12	395	45	-89	1310	2.28	2260	2.55
26 (15)	Cal. Spec.	16.4	12,000	388	4.6	1.0	40	290	3	-99	2310	2.94	2870	4.05
27 (24)	Ethyl Cellulose	16.5	1,600	48	9.2	0.5	2	45	1	-98	2410	3.2	4600	5.27
28 (22)	Alkyd	16.9	7,300	40	4.6	1.0	10	135	8	-94	1130	1.6	2000	2.04

TABLE II
HOT MELT ALKYD MODIFICATIONS

<u>Basis Formulation</u>	
	<u>% by wt.</u>
Alkyd (VBR-5010)	10
Staybelite #10	10
Pentalyn K	5
Small Glass Beads	25
Marble, RO-40	21½
Marble, XO	25
TiO ₂ -R-610	<u>3½</u>
	100

Additives

(Replacing 10% of Composite Vehicle in Basis Formulation)

<u>Hot Melt No.</u>	
36	Pentalyn K
37	Stabilite #10
38	Alkyd (VBR-5010)
40	Estane (Polyurethane Rubber)
41	Maleic Modified Ester (VBR-201)
43	Eastbond M-5 (Amorphous Polypropylene)
44	Ethylcellulose
45	Epon 1007
46	Pliolite AC
47	Abitol
49	Polyamide (Versamid 900)
50	SAIB (Eastman Chemical Company)
51	Sulfur (Stauffer)
52	Polycarbonate (Lexan - General Electric)

TABLE III
EFFECTS OF VEHICLE ADDITIVES

Hot Melt No.	Description	Density (wt./gal.)	Consistency		Elastic Modulus (psi x 10 ²)	Flexural Creep Properties		Static Shear Bond Strength			Flexural Impact Tests			
			@ 350°F	Slope @ 350°F		Initial Elastic Deformation	Creep Rate	@ 77°F	@ 0°F	Change	Max. Fiber Stress @ 77°F	Absorbed Energy @ 77°F	Max. Fiber Stress @ 0°F	Absorbed Energy @ 0°F
			(cps)	(cps/°F)		(in./in. x 10 ⁻³)	(in./in./min. x 10 ⁻³)	(psi)	(psi)	(%)	(psi)	(in.lb./ in. ²)	(psi)	(in.lb./ in. ²)
39	Basic Formulation	17.1	7,000	260	2.3	2.0	60	296	88	-70	590	1.17	1410	1.34
36	Pentalyn K	17.5	12,500	250	3.7	1.3	6.0	388	75	-81	618	1.06	618	0.82
37	Staybelite #10	17.5	10,500	410	3.7	1.3	50	400	282	-30	410	0.78	1180	1.48
38	Alkyd	17.1	16,500	390	0.74	6.3	600	412	150	-64	89.4	0.82	774	1.0
40	Estane	17.7	15,000	400	1.8	2.5	600	200	372	+86	956	2.43	1370	1.48
42	Basic Formulation	16.8	5,800	184	4.6	1.0	60	405	225	-44	266	0.72	1330	1.40
41	Mal. Mod. Ester (VBR-201)	17.6	15,000	920	9.2	0.5	6.0	400	120	-70	488	1.0	976	1.04
43	Eastbond M-5	17.2	21,000	860	9.2	0.5	70	325	285	-12	796	1.67	1500	1.72
44	Eth. Cellulose	17.0	34,000	1000	4.5	2.0	10	370	12	-97	1000	1.36	1590	1.70
45	Epon 1007	17.2			3.7	1.3	4.0	30	0	-100	1401	2.04	1500	1.58
46	Pliolite AC	17.5	26,000	1080	3.0	1.5	6.0	405	100	-75	860	1.08	1590	1.72
47	Abitol	17.8	9,500	230	1.8	2.5	100	412	240	-42	1095	1.30	1170	1.67
49	Polyamide (Versamid 900)	17.3	12,500	230	1.7	2.8	29	200	112	-44	1000	1.34	1410	1.44
50	SAIB	18.1	6,800	264	2.6	1.8	50	442	175	-60	860	1.03	1775	1.92
51	Sulfur	17.7	13,000	340	2.3	2.0	14	376	0	-100	1050	1.47	2120	2.4
52	Polycarbonate (Lexan)	17.7	6,200	716	6.2	0.8	2	335	150	-55	1095	1.10	1230	1.42

H.M. # 39 and H.M. # 41 is being investigated further, but it presently demands a very cautious interpretation of the tabulated results. The findings were somewhat disappointing to the extent that really substantial improvements in the original balance of the physical properties did not appear to be feasible. The capabilities of the Estane (polyurethane rubber) for improving low temperature bond was offset by an emanation of toxic vapors from this hot melt. Thus, service use of this composition would be impossible. In general, the polymeric additives yielded some enhancement in impact characteristics, but bonding characteristics were sacrificed prohibitively.

II. Test Development Work

A. Hot Melt Consistency

Complete temperature-consistency curves are of continuing interest for characterizing new materials; however, for routine comparisons of formulations we have adopted a two-parameter characterization. The primary parameter is consistency at 350° F. The secondary parameter is the slope of the temperature-consistency curve at 350° F which is reported in units of cps/°F. These parameters are tabulated for the hot melt formulations in Tables I and III.

B. Impact Tests

The flywheel impact tester was modified to accommodate the very low impact energy levels that characterize the hot melts. With the light-weight flywheel utilized, the total energy of the device was only seven inch-pounds. The tests reported in Tables I and III were determined from unnotched Charpy-type specimens $\frac{1}{2}$ " x $\frac{1}{2}$ " in cross-section on a span of 3.25 inches. Maximum fiber stress was computed from the impact load data in the same manner as for a static flexural strength test, thus

$$s = \frac{3PL}{2bd^2}$$

where: P = load, lbs
L = span, in
b = beam width, in
d = beam depth, in

Observed impact energy was converted to a basis of one square inch of specimen cross-section so the units of the test become in-lbs/in².

C. Elastic Modulus in Flexure, Elastic Deformation, and Creep

An approximate flexural elastic modulus was determined on the creep test device. The procedure involved loading a specimen with a fixed weight for about five seconds, observing the deflection, unloading, and immediately observing the recovery deflection. The difference in deflections is taken as the elastic deformation and

$$E_f = \frac{L^3}{4bd^3} \frac{P}{\delta}$$

where: E = modulus of elasticity in flexure, psi
L = span, in
b = beam width, in
d = beam depth, in
P = load, lbs
 δ = deflection difference, in

The creep characteristics of various formulations have been determined in the form of creep curves (deflection versus time at a fixed load). Similarly to consistency, these data have been condensed into two parameters for general characterization. The primary parameter is the Creep Rate which is taken arbitrarily as the slope of the deflection versus time curve at one minute. The Creep Rate is presented in units of

$$\frac{\text{in (deflection)}}{\text{in (gage length) min}} = \text{min}^{-1}$$

The secondary parameter is the instantaneous elastic deformation which represents the zero time intercept of the creep curve with the deflection ordinate. Its units are

$$\frac{\text{in (deflection)}}{\text{in (gage length)}}$$

One notes that the creep rate and elastic deformation are functions of the constant maximum fiber stress applied, whereas the elastic modulus is a property that is independent of the loading.

III. Inspection of Highway Applications

On December 3, 1965, the highway test applications placed on June 3, 1965, were inspected for integrity and night visibility and photographed. With the exception of a few incidences of random gouging (possible from rocks), and some very minor edge failure, the integrity of the hot melt stripes was unchanged. Night visibility of the hot melts was somewhat lower than on the previous inspection; however, the following hot melts were still above the 10 level: Permaline, #16 ($\frac{1}{2}$ Pliolite), #24 (Ethocel), #21 (Stikvel), #22 (Alkyd). The control paints were exhibiting definite wear (average integrity = 6, average night visibility = 7) and the superior durability of hot melts was clearly established.

Respectfully submitted,

W. R. Tooke, Jr.
Project Director

Approved:

W. H. Burrows, Head
Industrial Products Branch

GEORGIA INSTITUTE OF TECHNOLOGY

ENGINEERING EXPERIMENT STATION

ATLANTA, GEORGIA 30332

March 31, 1966



Division of Highway Planning
State Highway Department of Georgia
Atlanta, Georgia

Attention: Mr. H. H. Huckeba
State Highway Planning Engineer

Subject: Quarterly Progress Letter 5, Project A-802
"Hot Melt Traffic Marking Materials"
Contract No. 6401
Covering the Period from 1 January to 31 March 1966

Gentlemen:

Project activities during this quarterly report period were conducted in the following areas:

- I. Formulation Studies
 - A. "Conventional" Variations
 - B. Unextended High Polymers
- II. Inspection of Highway Tests
- III. Modification of Field Applicator

Details of these activities follow.

I. Formulation Studies

A. "Conventional" Variations

In the present context the term "conventional" refers to formulation studies which have centered about vehicles of the following general composition:

Conventional Formulation
of
Hot Melt Vehicle

<u>Component</u>	<u>Weight Per Cent</u>
High Polymer	15-40
Rosin Ester	30-70
Plasticizer	10-30

The vehicle of the California Specification is representative of this type.

Formulation objectives have been: (1) to increase the adhesion and (2) to improve impact resistance (toughness). Hopefully, we would seek to realize both objectives in a single material, but we would, if necessary, sacrifice one to maximize the other. The willingness to "trade off" adhesion is based on our observations that priming of concrete will restore adhesion satisfactorily. Moreover, priming of concrete is currently a standard field practice. On the other hand, we are interested in studying formulations of maximum inherent adhesion so that primers might be eliminated from the application process. Minimum requirements for toughness and adhesion must be established ultimately in the field.

Recent experimentation has demonstrated that use of about 15% of a solid epoxy resin (Epon 1001) in conventional formulations will very substantially improve adhesion without degrading other properties. Additionally, we have found that chopped glass fibers added to these formulations will enhance impact properties. Efforts to further enhance impact properties by the use of various high polymers have not been very fruitful. Ethyl cellulose and cellulose acetate butyrate impart definite superiorities in impact properties, but the thermal stability of these polymers is sub-marginal. At present we are deferring further work in the "conventional" formulations in favor of another approach as discussed below.

B. Unextended High Polymers

Recently we have found that certain polyamides and polyesters exhibit low enough melt viscosities to be useful as hot melt vehicles with only a minimum of plasticizing and fluxing additives. An outstanding example of results obtainable with a special polyamide (General Mills Versalon 1175) are shown in Table I. The extremely high absorbed energy shown by H.M. #99 is especially interesting, particularly in view of the low creep rates. Quantitative consistency data have not yet been obtained on these new items, but they are obviously in a workable range based on observed casting properties.

These results prove that very respectable strengths can be obtained in castable thermoplastic materials. Unless unanticipated shortcomings are observed, this could mean that very durable highway stripes might be realized at reduced film thicknesses. Formulation study is currently being concentrated in this area.

Aside from the application of this type material to traffic stripes, we feel that its special characteristics may suggest other unique uses in highway engineering and otherwise. For example, it might have interesting possibilities as a high strength but resilient grout, as a patching material, and for various bonding and joint filling applications. For any such applications we would not necessarily claim any outstanding performance characteristics for the thermoplastic, but rather we would emphasize the distinct advantages of very rapid and reliable set of a thermoplastic as opposed to the high sensitivity to environmental conditions and the relatively slow set of chemically cured resin systems. Since thermoplastic casting (as opposed to the usual injection or transfer molding operations) is novel to us as a feasible forming method for high polymers, we are

TABLE I
PHYSICAL TESTS OF UNEXTENDED HIGH POLYMERS

Hot Melt No.	Vehicle Components, Per Cent			Creep Rate (in./in./min. $\times 10^{-3}$)	Flexural Impact Tests at 77° F	
	Versalon 1175	Santicizer #8	Aroclor 1254		Max. Fiber Stress (psi)	Absorbed Energy (in.-lbs./in. ²)
96	100	-	-	0.2	7300	12.0
97	90	10	-	0.4	5840	30.8
98	90	-	10	0.2	8950	19.0
99	80	20	-	0.4	5820	50.7
100	80	-	20	0.3	7260	21.7
26	(California Specification)			40	2310	2.94

executing a Record of Invention on this work. A patent search will then be run to determine possible novelty.

II. Inspection of Highway Tests

Hot melt cross-stripe tests on I-85 were inspected, graded, and photographed on March 8, 1966 (9 months' weathering). The entire data file on this study has been transcribed to IBM card formats and is now accessible for any type of processing desired. We are exploring appropriate methods of data reduction, and anticipate that an analysis of variance may be particularly useful.

Qualitatively, the following observations can be made at 9 months' weathering:

1. As a group, the hot melts are greatly superior in film integrity and significantly superior in night visibility to good quality paints.
2. Priming significantly improves hot melts' integrity on concrete, and epoxy primer is superior to the rubber type. So little failure has been observed on asphalt, that a priming effect is not detectable.
3. Performance of hot melts on epoxy primed concrete is about equivalent to performance on unprimed asphalt--superior in both cases.
4. No significant effect of hot melt film thickness was observed--the thin films are as good as the thick ones.
5. As a group, the experimental hot melts were slightly superior to the proprietary control compositions. The exception was H.M. #19 which was high in consistency because of excessive vehicle polymer content. Its adhesion was obviously impaired.

These observations are very encouraging indeed, but do not justify complacency. We are tempted to conclude that erosion is not a significant problem except as it may affect bead retention and night visibility. Similarly, thin films appear to be completely satisfactory. Durability would appear to depend almost entirely on the ability of the coating to retain adhesion. Our earlier laboratory work has shown that adhesion of all of these field systems is deteriorated by thermal cycling. Thus we would continue to withhold judgment on the present field tests, and plan our second set for placement as soon as we have realized reasonable physical property goals which are now clearly in sight.

III. Modification of Field Applicator

Several minor modifications of our experimental hot melt applicator have been made to improve temperature control in the hot melt reservoir, and to facilitate uniformity of extrusion and thickness control. We are planning also to give our operator more "familiarization time" to

attain a degree of handling skill that was somewhat lacking in our previous applications. This unit will be ready for service during April.

Respectfully submitted,

W. R. Tooke Jr.
Project Director

Approved:

W. H. Burrows, Head
Industrial Products Branch

GEORGIA INSTITUTE OF TECHNOLOGY

ENGINEERING EXPERIMENT STATION

ATLANTA, GEORGIA 30332

July 8, 1966

Division of Highway Planning
State Highway Department of Georgia
Atlanta, Georgia

Attention: Mr. Emory C. Parrish
State Highway Planning Engineer

Subject: Quarterly Progress Letter No. 6, Project A-802
"Hot Melt Traffic Marking Materials"
Contract No. 6401
Covering the Period from 1 April to 31 June 1966

Gentlemen:

Project activities during this reporting period have been conducted in the following topical areas:

- I. Inspection and Analysis of Highway Tests, Series I
- II. Preparation and Placement of Highway Tests, Series II
- III. Field Wet Night Visibility Study

Details of these activities follow.

I. Inspection and Analysis of Highway Tests, Series I

This study was placed as cross-stripes on the outer north-bound lane of I-85 at the Lenox Road Bridge on June 1-3, 1965. The experimental design of the hot melt study encompassed the following variables:

	<u>Levels or Types</u>
Formulation	8
Substrate (concrete or asphalt)	2
Priming	3
Film Thickness	2

Two types of paint, alkyd and chlorinated rubber modified alkyd were also applied for comparisons. All details of the design and placements were discussed in Quarterly Progress Report No. 2 dated July 16, 1965.



These placements have been inspected for integrity and night visibility at quarterly intervals, and this report covers one full year of performance. In Table I one year observations of integrity (chipping - ASTM D913-51) and night visibility (ASTM D1011-52) are shown in factorial form together with averages resulting from data classifications chosen to display the main effects of the several independent variables. Note that individual observations are shown without decimals whereas averages are shown with two decimal places to emphasize the distinction.

A. Formulation

Integrities of the several formulations may be compared by examining an average of all observations on concrete and asphalt without primer for each formulation. Night visibilities were assumed to be independent of all variables except formulation, and thus may be compared as the means of all observations on a given formulation. A subjective "Application Rating" supplements the performance data. These results are tabulated below:

Relative Formulation Performance

<u>H.M. No.</u>	<u>Description</u>	<u>Average Integrity</u>	<u>Average Night Visibility</u>	<u>Average Application Rating</u>
108	Permaline	7.50	10.75	8.00
109	Cataphote	8.50	6.50	5.25
16	$\frac{1}{2}$ B/A Resin	8.25	11.50	6.62
15	Cal. Spec.	8.00	11.00	5.88
19	2 B/A Resin	6.50	4.50	4.25
21	Hydrocarbon	8.75	6.50	7.75
24	Ethyl Cellulose	7.50	10.00	8.75
22	Alkyd	9.00	8.50	7.25
Average		7.97	8.66	

We conclude that the general similarities rather than the differences among this series of diverse compositions is the most significant aspect of the formulation study. Even the absence of a distinct high polymer species (H.M. #21 and #22) does not greatly impair performance. The inclusion of excessive polymer (H.M. #19) may be more detrimental.

B. Substrate

A comparison of concrete versus asphalt, both without priming, yields from Table I the following average data:

Relative Substrate Performance, Integrity

Concrete	6.94
Asphalt	9.00

TABLE I
THERMOPLASTIC STRIPE OBSERVATIONS AT ONE YEAR, SERIES I

Thickness { H = 3/16" L = 3/32" Integrity/Night Visibility	#108 Permaline						#109 Catatherm					
	H		L		Ave.		H		L		Ave.	
	I	NV	I	NV	I	NV	I	NV	I	NV	I	NV
Concrete												
No Primer	7		4	10	5.50		9		6	8	8.00	
Rubber	8		5	11	6.50		9		5		7.00	
Epoxy	9		9		9.00		9		8		8.50	
Average	8.00		6.00		7.00		9.00		6.34		7.67	
Asphalt												
No Primer	9		10	11	9.50		9		9	5	9.00	
Rubber	9		9		9.00							
Epoxy	9		10		9.50							
Average	9.00		9.68		9.34							
Average Concrete and Asphalt												
No Primer	8.00		7.00		7.50		9.00		7.50		8.50	
Rubber	8.50		7.00		7.75							
Epoxy	9.00		9.50		9.25							
Average	8.50		7.83		8.17	(10.75)					(6.50)	

(Continued)

THERMOPLASTIC STRIPE OBSERVATIONS AT ONE YEAR, SERIES I

Thickness { H = 3/16" L = 3/32"	#16 S/A 1/2						#15 Calif. Spec.					
	H		L		Ave.		H		L		Ave.	
	I	NV	I	NV	I	NV	I	NV	I	NV	I	NV
Concrete												
No Primer	8		7	11	7.50		7		7	12	7	
Rubber	9		7		8.00		9		7		8	
Epoxy	10		9		9.50		10		10		10	
Average	9.00		7.67		8.33		8.66		8.00		8.33	
Asphalt												
No Primer	9		9	12	9.00		9		9	10	9.00	
Rubber												
Epoxy												
Average												
Average Concrete and Asphalt												
No Primer	8.50		8.00		8.25		8.00		8.00		8.00	
Rubber												
Epoxy												
Average					(11.50)						(11.00)	

(Continued)

TABLE I (Continued)

THERMOPLASTIC STRIPE OBSERVATIONS AT ONE YEAR, SERIES I

Thickness { H = 3/16" L = 3/32" Integrity/Night Visibility	#19 S/A 2						#21 Hydrocarbon Resin					
	H		L		Ave.		H		L		Ave.	
	I	NV	I	NV	I	NV	I	NV	I	NV	I	NV
Concrete												
No Primer	5		4		4.50		10		7		8.50	
Rubber	7	5	4		5.50		9		8		8.50	
Epoxy	10		10		10.00		10	7	10		10.00	
Average	7.33		6.0		6.66		9.66		8.33		9.00	
Asphalt												
No Primer	9		8	4	8.50		9		9	6		
Rubber												
Epoxy												
Average												
Average Concrete and Asphalt												
No Primer	7.00		6.00		6.50		9.50		8.00		8.75	
Rubber												
Epoxy												
Average					(4.50)						(6.50)	

(Continued)

TABLE I (Continued)

THERMOPLASTIC STRIPE OBSERVATIONS AT ONE YEAR, SERIES I

Thickness { H = 3/16" L = 3/32" Integrity/Night Visibility	#24 Ethyl Cellulose						#22 Alkyd					
	H		L		Ave.		H		L		Ave.	
	I	NV	I	NV	I	NV	I	NV	I	NV	I	NV
Concrete												
No Primer	8		5		6.50		9		8		8.50	
Rubber	10	10	9		9.50		9	9	6		7.50	
Epoxy	10		9		9.50		8		10		9.00	
Average	9.33		8.00		8.50		8.66		8.00		8.33	
Asphalt												
No Primer	9		8	10	8.50		9		10	8	9.50	
Rubber												
Epoxy												
Average												
Average Concrete and Asphalt												
No Primer	8.50		6.50		7.50		9.00		9.00		9.00	
Rubber												
Epoxy												
Average					(10.00)						(8.50)	

(Continued)

TABLE I (Concluded)

THERMOPLASTIC STRIPE OBSERVATIONS AT ONE YEAR, SERIES I

Thickness { H = 3/16" L = 3/32"	Formulation Averages					
	H		L		Ave.	
	I	NV	I	NV	I	NV
Integrity/Night Visibility						
Concrete						
No Primer	7.88		6.00		6.94	
Rubber	8.75		6.13		7.44	
Epoxy	9.50		9.38		9.44	
Average	8.71		7.23		7.94	
Asphalt						
No Primer	9.00		9.00		9.00	
Rubber						
Epoxy						
Average						
Average Concrete and Asphalt						
No Primer	8.44		7.50		7.97	
Rubber						
Epoxy						
Average						(8.66)

In the absence of priming treatments, integrity performance on asphalt was clearly superior.

C. Priming

Priming distinctly enhances integrity performance on concrete as indicated below:

Relative Priming Performance, Integrity on Concrete

No Primer	6.94
Rubber Primer	7.44
Epoxy Primer	9.44

Priming appears to have no effect when used on asphalt.

D. Film Thickness

From the Formulation Averages of Table I one notes that heavier films (nominal .188 in.) are clearly superior to lighter films (nominal .094 in.) on unprimed and rubber primed concrete, but the thickness effect is not significant on epoxy primed concrete or on asphalt.

E. Paint Comparisons

Paint observations will not be included in detail at this time. Performance averages at 12 months over concrete and asphalt for the two paints (alkyd and chlorinated rubber-alkyd) as compared with averages of unprimed hot melts are summarized as follows:

Paint Versus Hot Melts, 12 Months

	<u>Paints</u>	<u>Hot Melts</u>
Integrity	3.75	7.97
Night Visibility	3.75	8.66

Since several of the hot melt formulations are distinctly superior to the averages and since priming further enhances their performance, the comparisons shown are very conservative in representing the superiority of hot melts over painted stripes.

F. General Findings

Definite significant effects have been noted and itemized for the variables of formulations, substrate, priming, and film thickness of hot melts; and definite superiorities to paints were shown. The magnitudes of some of the differences must be considered as only tentative, subject to a further period of weathering. At this time, however, the useful life of hot melts is seen to be limited by the ability of these compositions to retain a satisfactory level of night visibility. Further research on night visibility retention of hot melts is clearly indicated.

II. Preparation and Placement of Highway Tests, Series II

A. Formulations

The experimental compositions selected for this study are detailed in Table II. Attention is concentrated on two variations of an alkyd based vehicle, and three variations of a polyamide based vehicle. The alkyd was chosen on the basis of materials economy and field proven performance; the polyamide was chosen because of outstanding properties exhibited in laboratory testing. This set of formulations provides a range of physical properties within an area of possible interest. It should improve our understanding of the relations of physical properties to service performance, and also provide a field test of specification candidates. The California Specification is included in the series as a control together with one proprietary hot melt yielding a total of seven hot melt materials for testing. In addition the alkyd and chlorinated rubber-alkyd paints were included in the tests as they were in Series I.

B. Experimental Design

The experiment was designed to explore the following variables:

Formulations	7
Substrates (concrete, asphalt)	2
Priming (none, epoxy-concrete only)	2
Surface Texture (none, corrugated)	2

Series I provided much guidance for design decisions. Applications to both concrete and asphalt substrates were definitely indicated. Epoxy had been found to be the preferred primer for concrete, but priming of asphalt was not indicated as useful. Film thickness was fixed at the level of 3/16 inch gate clearance, corresponding to the heavier applications of Series I. Surface corrugation of hot melts was investigated to determine if this method of enhancing wet night visibility would affect durability.

In summary, the experimental design was a four-factor factorial study with the epoxy primed asphalt treatment combinations missing. Randomization of line placement was also practiced in this study to minimize effects of unknown variables.

C. Highway Placements

Series II stripes were placed adjoining those of Series I on Interstate 85 on May 30-31, 1966. The randomization of placements included the paints as well as the hot melts. Individual stripes were identified as lines 401 through 424 on concrete and 425 through 441 on asphalt. Corrugations were applied using the "corrugator wheel" as shown attached to the hot melt applicator in Figure 1. Difficulty was experienced when some formulations tended to stick to the wheel lugs, so that optimum corrugations were not attained on many of the lines. Further development work on the corrugator is indicated.

TABLE II
FORMULATIONS OF HIGHWAY SERIES II

Hot Melt No. and Description	Composition in Per Cent by Weight					
	Cal. Spec.	Alkyd, LC	Alkyd, HC	Polyamide, HC	Polyamide, MC	Polyamide, LC
<u>Vehicle</u>	160	162	163	157	155	158
Abitol	5.79					
Pliolite	8.99					
Stabilite #10	9.79	8.92	10.32	7.38	12.29	18.43
VBR 5010		8.92	8.84			
Pentalyn K		6.71	5.39			
Polyniid 1560				12.90	9.21	4.66
Soutacizer 8				4.30	3.08	1.48
<u>Filler</u>						
Standard glass beads	20.78					
Fine glass beads				25.14		
TiO ₂ -R610	13.09			3.50		
RO-40 (CaCO ₃)	41.56			21.64		
XO (CaCO ₃)				25.14		

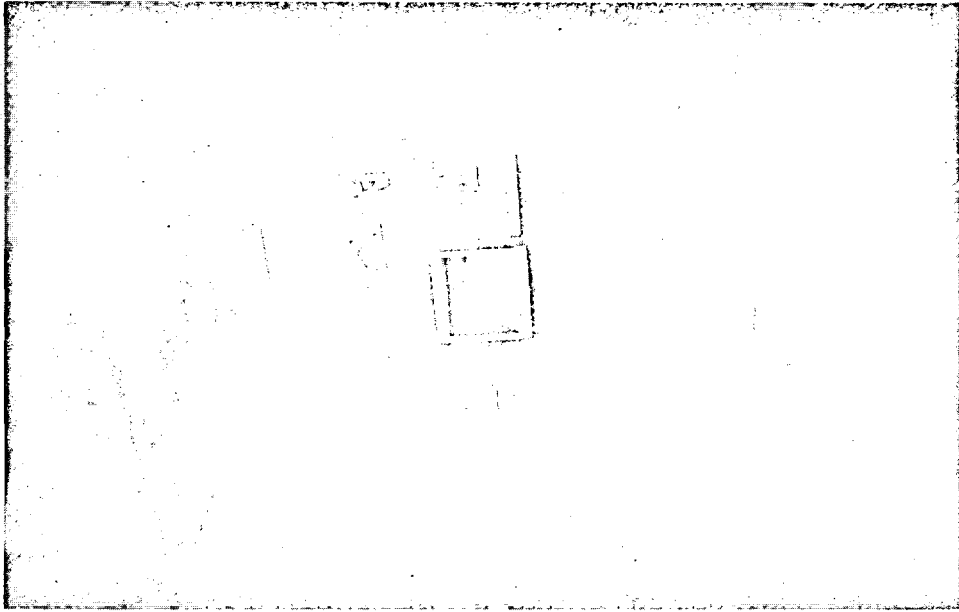


Figure 1. Corrugator Wheel Attached to Hot Melt Applicator.

The experimental hot melts were all placed satisfactorily. We did note, however, that hot melts #155 and #157, both containing relatively high levels of polyamide resin, were somewhat inferior in flow characteristics and were very slow in hardening after application. These same formulations were also most troublesome in sticking to the corrugator lugs. We anticipated that highway traffic might smear these lines, but this was not noted when traffic was released. Accommodations to the application difficulties can probably be made if these materials exhibit outstanding durability.

III. Field Wet Night Visibility Study

This study was undertaken to obtain comparative data on the wet night visibility characteristics of painted traffic stripes, plain hot melt stripes and corrugated hot melt stripes. In addition, it was hoped that the retention of wet night visibility under highway conditions might be assessed. Two sets of field tests were placed. The first set consisted of eight longitudinal stripes placed on Interstate 85. The second set involved only three stripes placed on the north edge of the Georgia Tech E.E.-Architecture parking lot. In both cases only one hot melt and one paint were used.

A. Highway Placements

This set was placed on May 31, 1966, on I-85 in the outer northbound lane between North Druid Hills Road and Clairmont Road. The lines were placed as a series of pairs, each running parallel to the center line of the highway and positioned one and two feet to the right of the center line. The experimental arrangement is summarized in Table III. The purpose of this study was to observe visually and photographically the relative retention of wet night visibility by these materials. It is hoped that this set can be photographed at an early date under wet night conditions. Optimum texturing was not attained, however, so that the results of this study cannot be considered as fully indicative of the potentials of a texturing treatment.

B. Parking Lot Placements

On June 3, 1966, a set of three parallel lines, each 15 feet long were placed on the north end of the parking lot between the E.E. and Architecture Buildings at Georgia Tech. The left line was plain H.M. #160, the center line was corrugated H.M. #160, and the right line was alkyd paint #95. The pavement was uniformly flat in this area, but sufficiently pitched to preclude standing water.

On the evening of June 17, 1966, at 9:30 PM these stripes were photographed under headlight illumination with a 16 mm movie camera equipped with a telephoto lens. Ranges of 350, 250, 150, and 50 feet were recorded. The stripes were then hosed with water and additional frames were recorded at the 50 foot range. Reproductions of the results are shown in Figure 2. We should emphasize that the apparent curvature of the center line is an actual slight curvature rather than a hump. At all ranges the center line is obviously superior in visibility in the

TABLE III
NIGHT VISIBILITY HIGHWAY PLACEMENT PLAN

<u>Line No.</u>	<u>Material</u>	<u>Hot Melt Texture (Plain or Corrugated)</u>	<u>Position</u>
442	H.M. #158	C	1
443	Paint #95		2
444	H.M. #158	P	1
445	Paint #95		2
446	Paint #95		1
447	H.M. #158	P	2
448	Paint #95		1
449	H.M. #158	C	2
450	H.M. #158	P	1
451	H.M. #158	C	2
452	H.M. #158	C	1
453	H.M. #158	P	2

dry state. In the wet state, as shown in 2d, it is the only line visible. The other lines are not deeply inundated. All lines are wetted with a thin film of water which completely quenches retroreflection at low angles of incidence against the horizontal surfaces of the plain lines, but only partially quenches the curved surfaces of the corrugated line that are angled toward the source.

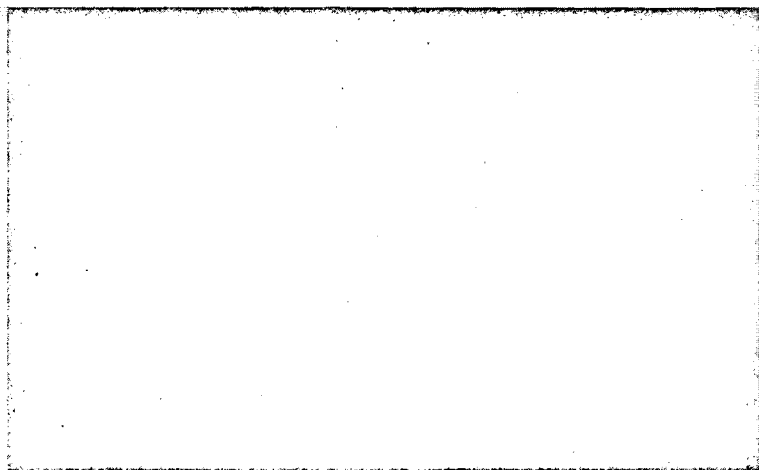
We conclude that the corrugation of hot melt traffic stripes is of definite practical interest as a means of enhancing night visibility, particularly under wet weather conditions.

Respectfully submitted,

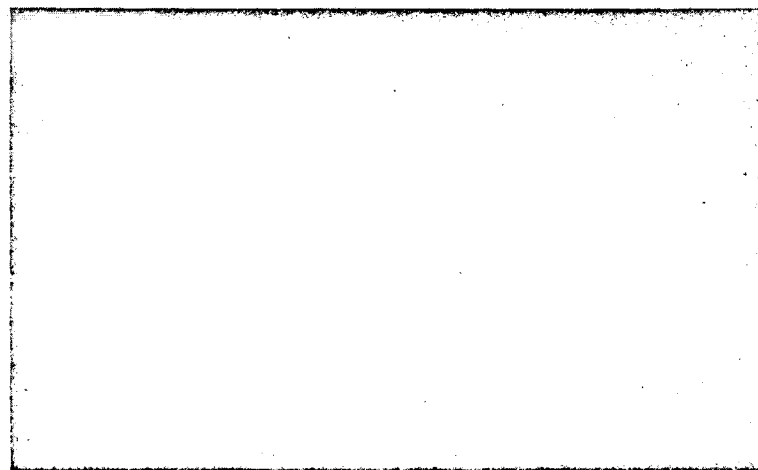
W. R. Tooke, Jr.
Project Director

Approved:

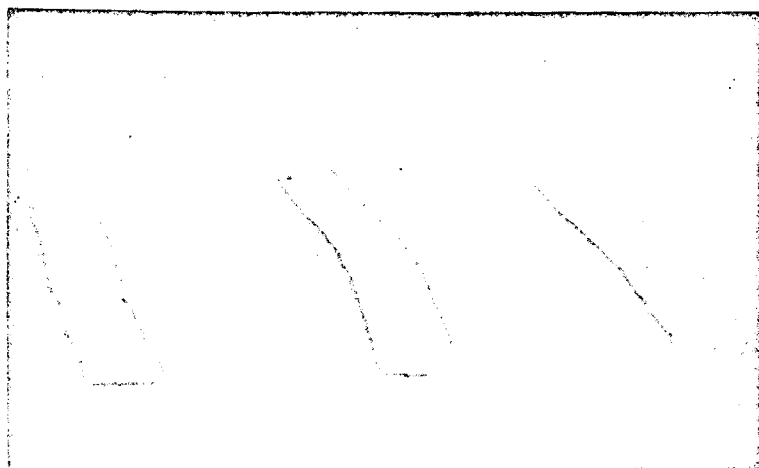
Frederick Bellinger, Chief
Chemical Sciences and Materials Division



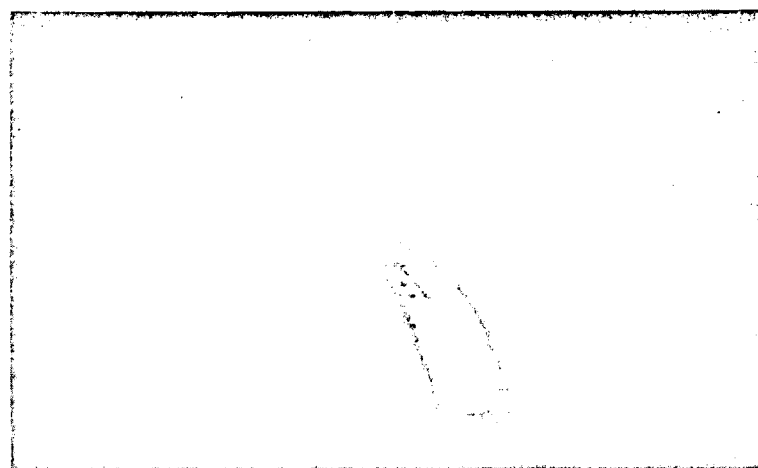
(a) RANGE 250 FT., DRY.



(b) RANGE 150 FT., DRY.



(c) RANGE 50 FT., DRY.



(d) RANGE 50 FT., WET.

Figure 2. Night Visibility Photographs.

GEORGIA INSTITUTE OF TECHNOLOGY**ENGINEERING EXPERIMENT STATION****ATLANTA, GEORGIA 30332**

October 12, 1966



State Highway Department of Georgia
2 Capitol Square, S. W.
Atlanta, Georgia 30302

Attention: Mr. Leland S. Veal
State Highway Planning Engineer

Subject: Quarterly Progress Letter No. 7, Project A-802
"Hot Melt Traffic Marking Materials"
Contract No. 6401
Covering the Period from 1 July to 30 September 1966

Gentlemen:

Project work during the current reporting period has been conducted in the following topical areas:

- I. Physical Property Data, Series II Highway Tests
- II. Study of Hot Melt Heat Aging
- III. Development of Specification Tests
- IV. Current Highway Observations, Series I and II

Details of these activities follow:

I. Physical Property Data, Series II Highway Tests

Retained samples of the hot melt formulations utilized in Series II highway tests were subjected to the same set of physical tests that were described previously for Series I.* The resulting data are presented in Table I. The unique properties of polyamides #155 and #157 are very apparent. They are characterized by relatively large elastic deformations, high bond strengths at 0° F, and exceptionally high impact properties. We believe the elasticity accounts for the mediocre application characteristics of these materials. Polyamide #158 contains a much lower quantity of the polyamide resin, and one notes that most of the physical properties are compromised accordingly.

We are unable to account for substantial differences in the properties of the California Specification #160 and Catatherm #164 as compared with Series I. The former was prepared by us from different batches of the

* Quarterly Progress Letter No. 4, Project A-802, dated January 10, 1966.

TABLE I
PHYSICAL TESTS OF HIGHWAY TEST COMPOSITIONS,* SERIES II

Hot Melt No.	Description	Density wt/gal	Consistency		Elastic Modulus (psiX10 ⁵)	Flexural Creep Properties		Static Shear Bond Strength			Flexural Impact Tests			
			50° F (cps)	Slope @ 350° F (cps/°F)		Initial Elastic Deformation (in/inX10 ⁻³)	Creep Rate (in/in/minX10 ⁻³)	@ 77° F (psi)	@ 0° F (psi)	Change (%)	Max. Fiber Stress @ 77° F (psi)	Absorbed Energy @ 77° F (in lb/in ²)	Max. Fiber Stress @ 0° F (psi)	Absorbed Energy @ 0° F (in lb/in ²)
155	Middle Polyamide	16.39	7,000	260	0.35	13.0	150	0	500 ⁺	-	5,990	15.3	2,680	4.2
157	High Polyamide	16.61	8,000	920	0.21	22.0	35	225	500	+120	-	> 77	3,400	4.5
158	Low Polyamide	16.3	5,000	180	1.8	2.5	35	400 ⁺	100	-75	1,770	2.6	2,040	2.5
160	Cal. Spec.	16.7	4,000	720	4.6	1.0	135	380	340	-10	2,500	3.4	4,000	4.8
162	Alkyd	17.0	3,000	360	4.6	1.0	32	285	20	-93	1,230	2.0	2,220	3.2
163	Alkyd	17.0	4,000	480	2.3	2.0	390	250	150	-40	1,360	2.7	2,040	2.8
164	Catatherm	18.1	2,000	1160	4.6	1.0	260	290	300	+03	2,320	3.5	2,270	3.0

* These samples were drawn from molten hot melts at the time they were being applied to the highway.

same raw materials. Possibly one or more materials was sufficiently different to alter the properties. The latter was ordered from the manufacturer as the same material but may be, in fact, a different formulation. In any case, we are forced to conclude that these formulations cannot be treated as reference standards common to the two test series.

II. Study of Hot Melt Heat Aging

A tendency of most hot melts to degrade in color and physical properties upon prolonged heating has been observed from the beginning of our formulation work. This factor has often been suspected as an important source of difficulties in reproducing physical test results. Accordingly, a decision was made to study the heat aging effect and to consider the advisability of standardizing a heat aging treatment as a preliminary to physical testing so that the observed properties of hot melts would reflect their thermal stability.

Treatment conditions were chosen that were believed to be realistic with respect to current field practices.

Heat Aging Conditioning

Temperature	-	400 \pm 10° F
Time	-	4 hours
Agitation	-	Slow, continuous, approx. 250 RPM

The material for treatment is melted in a heated 1 quart stainless steel beaker with a propeller agitator that is set close to the bottom. The beaker should be at least half filled with molten material. Timing begins when the specified temperature is reached. Test specimens should be prepared immediately following conditioning.

Physical test changes for several formulations before and after heat aging are given in Table II. The California Specification was found to be very stable in consistency, catatherm increases substantially, but all other hot melts are degraded to various degrees. Flexural creep tends to be reduced by heat aging except for the alkyds. Since the alkyds contain no volatile plasticizers, they cannot be stiffened by plasticizer losses, so they must be "self-plasticized" by alkyd degradation. Heat aging produces losses in impact properties in most cases. Catatherm is again an outstanding exception - possibly this material undergoes some polymerization or condensation.

Regardless of the causes, significant changes of properties occur in all hot melts as a result of heat aging. Service application of hot melts may be expected to involve varying degrees of heat aging. Therefore, a standardized heat aging conditioning is an appropriate preliminary step for the characterization of hot melt compositions.

TABLE II
EFFECTS OF HEAT AGING

Hot Melt No.	Description	Consistency @ 350° F Change (%)	Flexural Creep Change (%)	Flexural Impact	
				Max. Fiber Stress @ 77° F Change (%)	Absorbed Energy @ 77° F Change (%)
155	Middle Polyamide	-4	0	-42	-81
157	High Polyamide	-19	-90	+26	-99
158	Low Polyamide	-24	-50	-11	-10
160	Cal. Spec.	0	-29	-34	-24
162	36% Alkyd	-39	+3000	+11	+6
163	42% Alkyd	-7	+36	-46	-20
164	Catatherm	+98	-38	+31	+15

III. Development of Specification Tests

Accumulating experience indicates that hot melt compositions exhibiting satisfactory field durability can be prepared from numerous types of raw materials. The development of specifications requiring particular resin and plasticizer ingredients would appear to be unwise at the present time for several reasons. A sufficient reason is that the "state of the art" has not yet been sufficiently developed that a specification of compositional limitations can be adequately supported by materials characterization methods and by field performance findings. Moreover, the use of a compositional specification would tend to discourage formulation research by suppliers and potential suppliers. Such research is needed. Most important, we feel that the requirements for hot melt traffic marking materials can be defined adequately by properly chosen laboratory performance tests. These requirements can be advanced, as desired, with advances in the state of the art so as to encourage maximum ingenuity in achieving improved performance and economy.

Performance specification testing is being approached from a different point-of-view from much of the research and development testing pursued earlier. Research testing has involved efforts to determine functional relationships of composition and physical properties. Specification testing is aimed at establishing minimum acceptable performance levels. In some cases equipment and procedures can be simplified, but at the same time,

increased attention is being directed to environmental conditioning and cycling procedures to assure adequate retention of properties under anticipated field weathering conditions.

For example, all hot melts for specification testing are first subjected to the heat aging conditioning previously described. Specimens for impact tests and bonding tests are also subjected to a period of water immersion, freezing, and thawing under water. Replicates are tested in the frozen condition and after thawing. This conditioning is extremely destructive, and appropriate allowances must be made for substantial losses in performance for even the best formulations.

Various details of conditioning, cycling environment, and test methods are being examined currently for the purpose of evolving adequate performance specification procedures with a minimum of complexity and redundancy.

IV. Current Highway Observations, Series I and II

At 15 months, Series I stripes show some further deterioration from the 12 months' observations. The average integrity reduction was about one unit with the exception of Hydrocarbon Resin #21 which went down three units and showed severe cracking. Several systems are still almost perfect in integrity, although some further decline of night visibility has occurred. In Series I the paint controls were already below an integrity-4 average at 12 months. At 15 months, further slight deterioration was noted.

Of the Series II stripes at 3 months, all polyamides (#155,157,158) rated integrity-10, the others (#160, 162, 163, 164) averaged integrity-8, and paints averaged integrity-7. Among the corrugated stripes some "flattening" was evidenced in all cases, but it was almost negligible with the polyamides. Catatherm #164 exhibited a slight widening of the stripe in the "tire ruts."

As reported previously, all hot melt test stripes of Series I and II on asphalt were destroyed by repaving operations. Fortunately, we have seen that P/C concrete imposes a more severe performance test and the monitoring of these stripes is continuing.

Respectfully submitted,

W. R. Tooke, Jr.
Project Director

Approved:

Frederick Bellinger, Chief
Chemical Sciences and Materials Division

A-802

GEORGIA INSTITUTE OF TECHNOLOGY

ENGINEERING EXPERIMENT STATION

ATLANTA, GEORGIA 30332

January 12, 1967



State Highway Department of Georgia
2 Capitol Square, S. W.
Atlanta, Georgia 30302

Attention: Mr. Leland S. Veal
State Highway Planning Engineer

Subject: Quarterly Progress Letter No. 8, Project A-802
"Hot Melt Traffic Marking Materials"
Contract No. 6401
Covering the Period from 1 October to 31 December 1966

Gentlemen:

Project work for this reporting period has included the following topics:

- I. Specification Test Development
- II. Drafting of Hot Melt Specifications
- III. Outline of Technical Report

Details of these activities follow:

I. Specification Test Development

As discussed in previous reports, we have undertaken to develop hot melt specifications based on physical, environmental, and performance tests rather than on compositional recipes.

Initial physical properties can provide only very limited guidance as to performance capabilities. A material must be subjected to appropriate environmental stresses before its properties may be directly related to service performance. We have selected the following conditioning procedures to meet this requirement:

- 1. Heat Aging Treatment
- 2. Water-Freezing Cycle Treatment

Details of these treatments and other tests are attached.

It was proposed that for purposes of specification testing, all hot melts be subjected first to the Heat Aging Treatment. Test specimens would be prepared only from the heat-aged material. In this condition, the following tests would be performed:

1. Viscosity
2. Flexural Creep Rate
3. Static Indentation
4. Reflectance

Other specimens would be subjected to the Water-Freezing Cycle Treatment for the following tests:

1. Static Tensile Bond
2. Falling Ball Impact

It will be noted that these tests are performed on (a) frozen specimens at 0° F, and (b) frozen, thawed and water soaked specimens at 77° F.

A portion of the experimental work in support of this testing scheme is summarized in Table I. By comparisons of "As Received" versus "Heat Aged" specimens, one notes that heat aging produces variable but often very significant effects on viscosity, creep, and static indentation; however, only the properties in the heat-aged condition are judged to be relevant to specifications. Present judgments regarding acceptable levels of the test results are indicated at the bottom of Table I and in the specification draft discussion. The tabulation clearly shows some of the destructive effects of the water freeze cycle on tensile bond and impact. Fairly good bonds are the rule in the frozen condition, but subsequent thawing seriously disrupts bond integrity. On the other hand, impact resistance is reduced by freezing, but recovers upon thawing.

Additional specification testing development work is being pursued as the results of highway cross-stripe tests mature and service performance-property correlations can be examined.

II. Drafting of Hot Melt Specifications

Some rather broad specification limits have been indicated at the bottom of Table I. These limits may be modified and additional tests introduced as more information is accumulated. A summary discussion of specific items follows:

Viscosity. Within the broad limits indicated, viscosity is not a critical variable. Since we have observed poor application characteristics at low viscosities and good application characteristics at high viscosities (within the specified range), other rheological parameters are involved. The polyamides, for example, exhibit marginal application characteristics even at very low viscosities. Flow behavior is probably complicated by the presence of elasticity and yield stresses. A comprehensive study of hot melt rheology

TABLE I
SPECIFICATION DEVELOPMENT TESTS

									Water-Freezing Cycle			
		"As Received"			Heat Aged					Falling Ball Impact		
										@ 77° F	@ 0° F	
Hot Melt No.	Description	Consistency @ 350° F (cps)	Flexural Creep Rate (in./in./min x 10-3)	Static Indentation 200 gm (mils)	Consistency @ 350° F (cps)	Flexural Creep Rate (in./in./min x 10-3)	Static Indentation 200 gm (mils)	Reflectance (Green filter)	Static Tensile Bond @ 77° F (psi)	@ 0° F (psi)	(Per cent unbroken after 3 impacts)	(Per cent unbroken after 3 impacts)
155	Middle Polyamide	4,600	3.0	47	4,400	3.0	78	53	200+	200+	100	100
157	High Polyamide	3,700	250	120	3,000	24	62	56	200+	200+	100	100
158	Low Polyamide	3,400	4.0	47	2,600	2.0	78	52	17	78	100	20
160	Calif. Spec.	26,500	7.0	140	26,500	5.0	110	76	15	61	100	100
162	Alkyd	11,400	4.0	190	7,800	130	510	37	26	45	60	20
163	Alkyd	7,500	35	230	7,000	55	470	49	2	56	100	0
164	Catatherm	9,000	48	260	17,800	30	310	73	25	96	100	40
169	Calif. Spec. Special	25,000	2.0	62	21,500	2.0	78	73	43	96	-	-
Tentative Specification Limits:												
Maximum					35,000	30	200	-	-	-	-	-
Minimum					2,000	-	-	74	-	-	80	80

would be very helpful, but is not immediately feasible. Accordingly, we shall probably be required to include a definition of satisfactory field application characteristics as a basic specification requirement.

Flexural Creep Rate. An upper limit on flexural creep is needed to preclude use of materials that will flatten and flow excessively in service. However, two or more creep grades may be established eventually to adapt to particular traffic and climatic requirements.

Static Indentation. Frequently this test will correlate with the flexural creep. However, the static indentation test detects creep effects under much lower loadings over an extended period of time. Excessive indentation also suggests probable bead retention or reflectivity problems.

Reflectance. The requirement for a satisfactory level of diffuse reflectance of heat-aged hot melt precludes the occurrence of excessive discoloration, and requires the presence of adequate white pigmentation.

Static Tensile Bond. This is a critically important test. Currently maturing field data must be examined, and further laboratory work undertaken to establish a valid procedure. At present, meaningful specification limits cannot be projected.

Falling Ball Impact. Excessive brittleness, particularly at low temperatures, is detected by this test. The results have been reliably consistent with other available data. The indicated specification limits are consistent with the capabilities of available practical hot melt compositions.

If the foregoing discussions indicate dissatisfaction with certain specification tests, this is indeed our present feeling. We sincerely solicit any new ideas, particularly on bonding tests. In the meantime, we are encouraged by the fact that a number of our highway placements in June 1965 are still exhibiting good integrity.

III. Outline of Final Report

Subject to revisions, the organization of the final report has been framed as follows:

Introduction

- State-of-the-Art
- Project Objectives

Exploratory Preparation and Characterization

Systematic Formulation Studies

- Filler Properties
- Vehicle Properties
- Composite Properties

Highway Cross-Stripe Testing

Equipment
Series I
Series II
Wet Night Visibility

Specification Development Work

Environmental Treatments
Suitable Acceptance Test Methods
Laboratory-Field Correlations

Appendix

Specifications
Test Methods

Respectfully submitted,

W. R. Tooke, Jr.
Project Director

Approved:

Frederick Bellinger, Chief
Chemical Sciences and Materials Division

Attachment

ENVIRONMENTAL TREATMENTS ON SPECIFICATION TESTS

Heat Aging Treatment

Temperature	-	$400 \pm 10^{\circ}$ F
Time	-	4 hours
Agitation	-	Slow, continuous, approx. 250 RPM

The material for treatment is melted in a heated 1 quart stainless steel beaker with a propeller agitator that is set close to the bottom. The beaker should be at least half filled with molten material. Timing begins when the specified temperature is reached. Test specimens should be prepared immediately following conditioning.

Water-Freezing Cycle Treatment

Samples are placed in a water bath maintained at 77° F for 18 hours. The samples are then transferred to a freezer maintained at 0° F for 3 hours. The samples to be tested at 0° F are tested as soon as they are removed from the freezer. The samples to be tested at 77° F are returned to the water bath for 2 hours and then tested.

Consistency at 350° F

Consistency of hot melts is measured using a Brookfield viscometer and an oil bath maintained at 350° F. The sample of approximately 800 g is placed in a Size 303 can, heated to a uniform temperature slightly above 350° F and placed in the oil bath. A Brookfield spindle is warmed and then placed in the sample and moved around to bring it to the temperature of the hot melt and release any air trapped under it before attaching it to the viscometer. The reading is taken when the temperature of the hot melt has reached 350° F. The sample should be carefully stirred just prior to taking the reading to insure uniformity.

Flexural Creep Test

The flexural creep of a hot melt is obtained by measuring the sagging displacement of a $1/2 \times 1/2 \times 6$ in. bar of hot melt which is suspended over supports 4 inches apart, and a load of 3550 grams applied to the center of the bar. The sagging is measured by a dial micrometer.

The mils of sagging are plotted against time on linear graph paper and the slope at one minute is taken as the flexural creep.

Static Indentation Test

The static indentation is obtained by measuring the diameter of the impression left by a $3/4$ in. diameter steel ball loaded to 200 grams for 24 hours on a hot melt disk prepared as described in the Falling Ball Impact Test.

Tensile Bond Strength

Specimens for the tensile bond strength of a hot melt are obtained by cementing two $3 \times 5 \times 3/4$ in. concrete blocks together with a $2 \times 2 \times 1/4$ in. layer of hot melt at 350° F in such a manner that the long dimensions of the blocks are at 90° to each other. Before the cementing operation the faces of the concrete blocks are sandblasted to remove laitance. The samples are subjected to the Water-Freezing Cycle as a part of the testing procedure. Specimens are tested in a hydraulic press for tensile bond strength.

Note: The sample should be placed in the press in such a manner that the only forces on the bond are tensile forces.

Falling Ball Impact Test

Prepare 10 disks from heat-aged hot melt which are 0.25 ± 0.025 in. thick and approximately 2 in. in diameter.* These disks should not vary more than $\pm 5\%$ in weight. The disks are subjected to the water-freezing cycle as a part of the testing procedure.

A disk is placed on an anvil which is a 3 pound block of brass 2 in. in diameter with the top covered by a $1/16$ in. layer of rubber.

A $1/2$ in. diameter steel ball is dropped onto the disk from a position one foot above and centered on the disk. Failure is considered to have occurred when a crack appears in the sample. Four of the five disks must withstand three drops of the steel ball without failure for the material to pass.

* Aluminum Foil Cups, Fisher No. 8-732, are satisfactory for molds.

GEORGIA INSTITUTE OF TECHNOLOGY

ENGINEERING EXPERIMENT STATION

ATLANTA, GEORGIA 30332

April 6, 1967

State Highway Department of Georgia
2 Capitol Square, S. W.
Atlanta, Georgia 30302

Attention: Mr. Leland S. Veal
State Highway Planning Engineer

Subject: Quarterly Progress Letter No. 9, Project A-802
"Hot Melt Traffic Marking Materials"
Contract No. 6401
Covering the Period from 1 January to 31 March 1967

Gentlemen:

Project work for this reporting period has included:

- I. Final Laboratory Testing
- II. Drafting Hot Melt Specifications and Final Technical Report
- III. Highway Observations, Series I and II

Details follow:

I. Final Laboratory Testing

Among the formulations of Highway Series I, the recently developed acceptance-type tests had not been run. This work was completed for formulations of interest so that comparable data are now available for both Series I and II. Details will appear in the final report, however, it was a noteworthy and pleasant surprise to discover that the ethyl cellulose based hot melt (# 24) exhibited very good thermal stability.

Acceptance testing procedures were described in Progress Report No. 8. Photographs of simple apparatus used in these tests are shown in Figures 1, 2, and 3.

II. Specifications and Report Drafting

The final report is now in crude draft form and current attention is directed toward specifications. A draft of specifications for "Clear Primer for Hot Melt Traffic Lines" is attached.

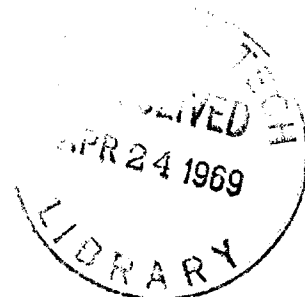




Figure 1. Static Indentation Test.

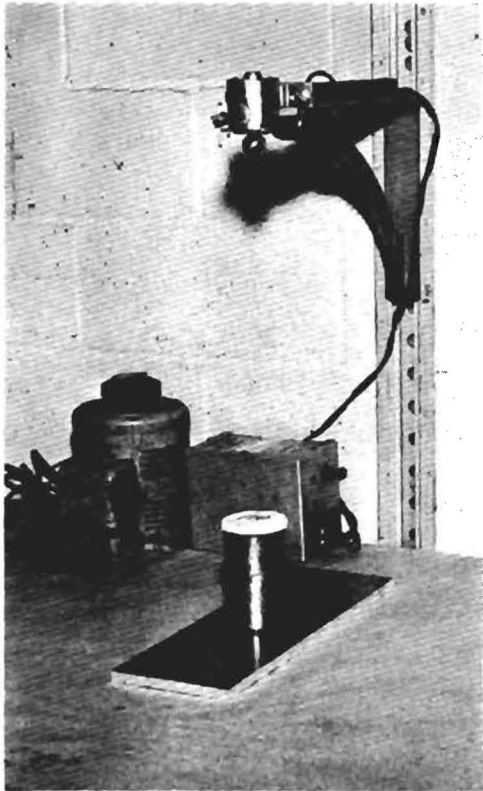


Figure 2. Falling Ball Impact Test.

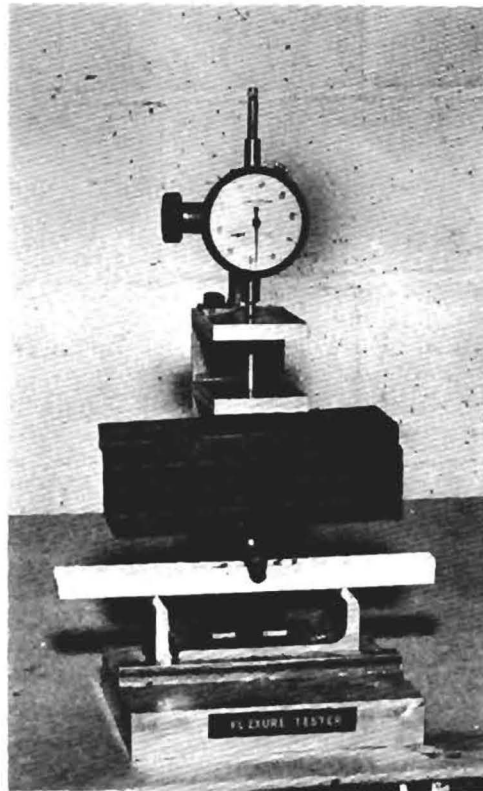


Figure 3. Flexure Test.

III. Highway Observations, Series I and II

On January 3, 1967, all highway placements were examined for integrity and night visibility. Series I had 19 months' service and Series II had 7 months' service.

Average readings of integrity and night visibility for the various formulations of Series I are presented in rank order in Table I. In addition, the effects on integrity of priming, hot melt thickness, and thickness-priming interactions are shown.

Data from Series II are summarized in Table II. The overall average effect of epoxy priming on integrity is shown. (Only epoxy and standard H.M. thickness were involved in this study.) The relatively poor average performance of the polyamides reflects catastrophic adhesion failures on unprimed concrete - where epoxy primer was used, integrity performance was superior.

Current project planning is pointed toward concluding the final report as soon as feasible following June observations of highway stripes.

Respectfully submitted,

W. R. Tooke, Jr.
Project Director

Approved:

Frederick Bellinger, Chief
Chemical Sciences and Materials Division

Attachment

TABLE I
PERFORMANCE OF SERIES I, 19 MONTHS
(Ratings are based on averages of all stripes for each formulation type.)

<u>H.M. No.</u>	<u>Description</u>	<u>Integrity</u>	<u>H.M. No.</u>	<u>Description</u>	<u>Night Visibility</u>
22	Alkyd	8.50	-	Permaline	12
24	Ethyl Cellulose	8.17	16	$\frac{1}{2}$ S/A Resin	10
-	Catatherm	7.33	-	Catatherm	9
16	$\frac{1}{2}$ S/A Resin	7.00	22	Alkyd	8
15	Calif. Spec.	6.16	24	Ethyl Cellulose	7
21	Hydrocarbon Resin	5.67	15	Calif. Spec.	7
-	Permaline	5.33	21	Hydrocarbon Resin	6
19	2X S/A Resin	3.17	19	2X S/A Resin	5
Total Average		6.5			
Priming Effect:					
Overall Unprimed		5.5			
Overall Rubber Primed		6.3			
Overall Epoxy Primed		7.44			
H.M. Thickness Effect:					
Overall, 3/32"		5.92			
Overall, 3/16"		6.92			
Thickness-Priming Interactions:					
3/32", Unprimed		4.88			
3/32", Rubber Primed		5.63			
3/32", Epoxy Primed		7.25			
3/16", Unprimed		6.13			
3/16", Rubber Primed		7.00			
3/16", Epoxy Primed		7.63			

TABLE II

PERFORMANCE OF SERIES II, 7 MONTHS

(Ratings are based on averages of all stripes for each formulation type.)

<u>H.M. No.</u>	<u>Description</u>	<u>Integrity</u>	<u>H.M. No.</u>	<u>Description</u>	<u>Night Visibility</u>
160	Calif. Spec.	8.33	157	Polyamide	28
164	Catatherm	8.33	155	Polyamide	16
163	Alkyd	8.33	162	Alkyd	14
158	Polyamide	8.33	163	Alkyd	14
162	Alkyd	7.67	160	Calif. Spec.	13
157	Polyamide	5.67	158	Polyamide	12
155	Polyamide	4.33	164	Catatherm	10
Total Average		6.62			
Overall Unprimed		6.64			
Overall Epoxy Primed		8.57			

STATE HIGHWAY DEPARTMENT OF GEORGIA
TENTATIVE MAINTENANCE SPECIFICATION
FOR
CLEAR PRIMER FOR HOT MELT TRAFFIC LINES

General

This tentative specification was developed under Research Project No. A-802 (Contract No. 6401) based upon experimental findings and currently available technical information. It is requested that recommendations for changes be directed to the Engineer of Materials and Tests of the State Highway Department of Georgia.

No prior specifications have been issued covering the product and application described hereunder.

Scope and Classification

Scope - This primer is intended for use on Portland cement and asphaltic concrete surfaces prior to the application of thermoplastic hot melt traffic lines.

Classification - Clear primer shall be of the following types as specified in the invitation for bids, contract or order.

Type I - Synthetic rubber - resin type

Type II - Epoxy - polyamide type, two package,
Parts A and B

Requirements

Materials

Materials shall be as herein specified. Any materials not definitely specified shall be of good commercial quality.

Primer Composition

The primer components shall be as specified in Table I and below for the respective types of primer.

Type I - This primer shall be supplied in a single package, and shall consist of an intimate blend of Buna N rubber with a compatible phenolic resin in a solution of the specified solvents.

When dried upon a potassium bromide disk, a film of the primer shall have infrared absorption maximums at the same wavelengths and to the same relative degree as that shown by the curve of Figure 1.

TABLE I
PRIMER COMPOSITION
(Percent by weight of primer)

<u>Ingredients and Specifications</u>	<u>Type I</u>	<u>Type II</u>	
		<u>Part A</u>	<u>Part B</u>
Buna N rubber	6-8		
Phenolic resin	3-4		
Epoxy liquid resin		50 min.	
Polyamide resin			50 min.
Methyl ethyl ketone (TT-M-261)	45 max.		
Methyl isobutyl ketone (TT-M-268)	18 max.		
Diisobutyl ketone	4.5 max.		
Toluene (TT-T-548a)	18 max.		
Xylene (TT-X-916)	4.5 max.		
Acetone (ASTM D329-58)		50 max.	50 max.

Type II - This primer shall be supplied in two packages of approximately equal volume. Part A shall consist of a liquid epoxy resin, viscosity 150 poises max. (at 25° C), epoxide equivalent 175-210; a bisphenol-type resin exhibiting an infrared spectrum as shown in Figure 2. It shall be supplied in an acetone solution. Part B shall consist of a liquid polyamide resin, viscosity 7-9 poises (at 75° C), amine value 290-320 (mg. KOH/g sample); a dimerized fatty acid - aliphatic polyamine-type resin exhibiting an infrared spectrum as shown in Figure 3. It shall be supplied in an acetone solution.

Primer Qualitative Requirements

Condition in Container - In a freshly-opened full can the primer shall appear smooth and homogeneous with no skins or gel particles.

Storage Stability - When stored up to six months from date of delivery, the primer shall exhibit no appearance change as compared with initial condition in container.

Mixing Properties, Type II - When Parts A and B are mixed in equal volumes a smooth homogeneous mixture shall result. When the mixture is aged in a filled, closed container for eight hours it shall show no signs of gellation.

Spraying Properties - The primer shall spray satisfactorily in all respects, and show no signs of cobwebbing or dusting.

Sampling, Inspection, and Testing

Unless otherwise specified, sampling, inspection and testing shall be in accordance with Method 1031 of Federal Standard 141. Samples shall be submitted with each bid. Each sample shall consist of one-half gallon of primer submitted in quart cans (one quart each of Parts A and B, Type II).

Packaging

Clear primer for hot melt traffic lines shall be delivered in 55 gallon drums unless otherwise specified. The description, requisition number, purchase order number, gross and net weights shall be stenciled on the head of each drum. The drums shall be of the removable head type.

Certified Analysis

The manufacturer shall submit with his bid sample a certified analysis setting forth the composition of the primer. It shall include the following information: Percentage composition of the primer to include brand and kind of ingredients. It shall contain the actual test data obtained by the manufacturer in determining compliance with the physical tests required by these Specifications. In addition, the manufacturer shall further certify that if awarded the contract the material furnished will be identical with that of the sample submitted. This information shall be held as confidential.

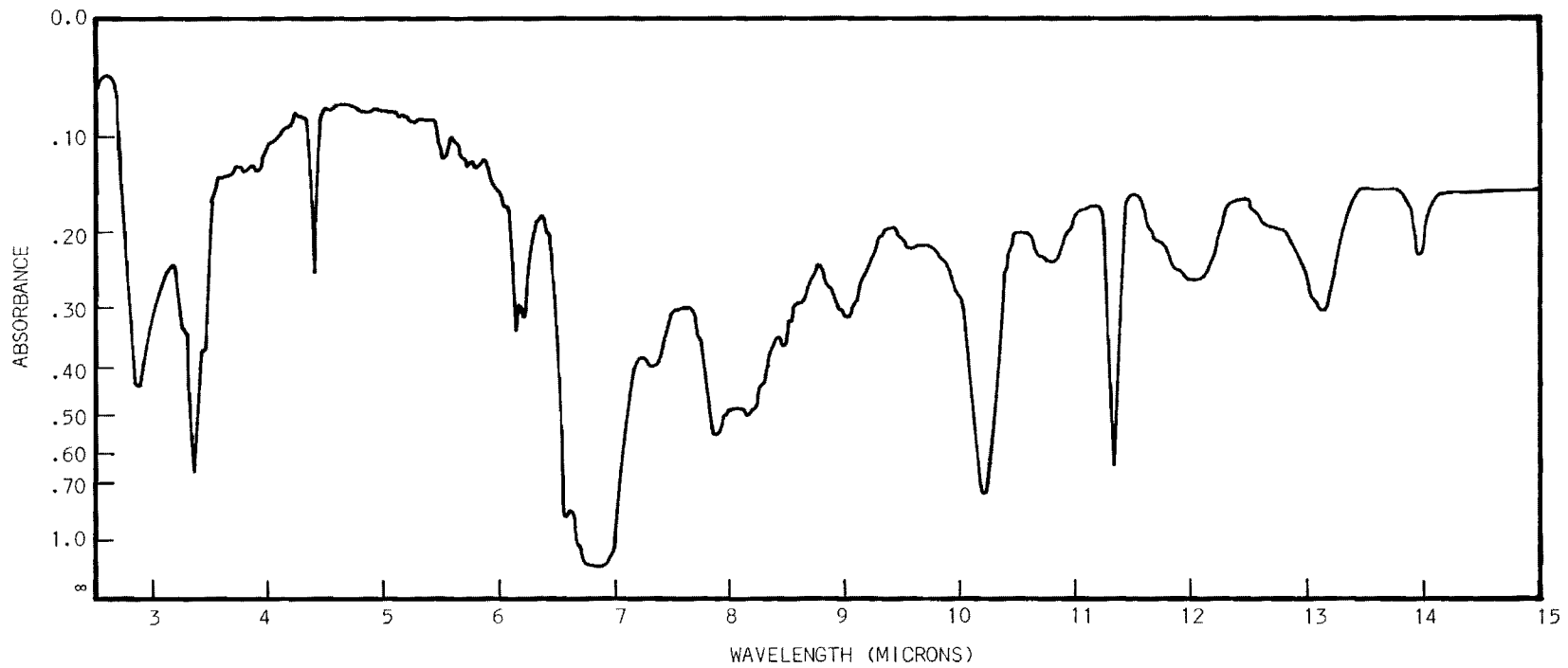


Figure 1. Buna N - Phenolic Resin Infrared Spectrum

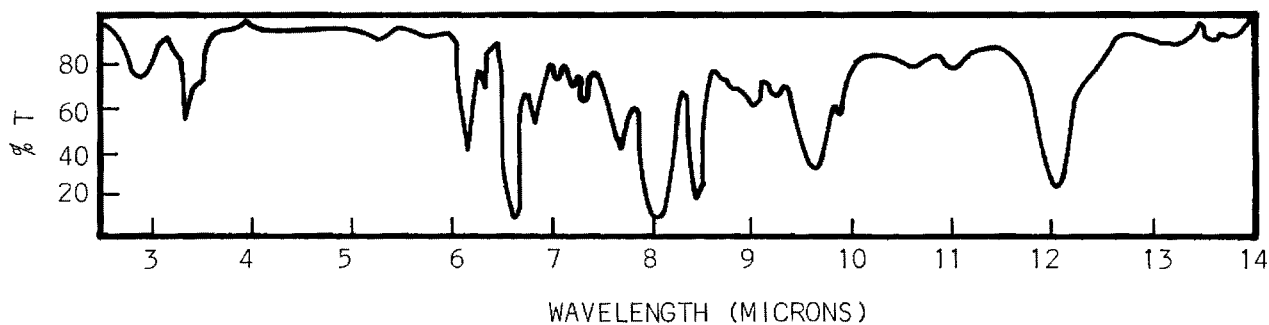


Figure 2. Bisphenol Type Epoxy Resin Infrared Spectrum

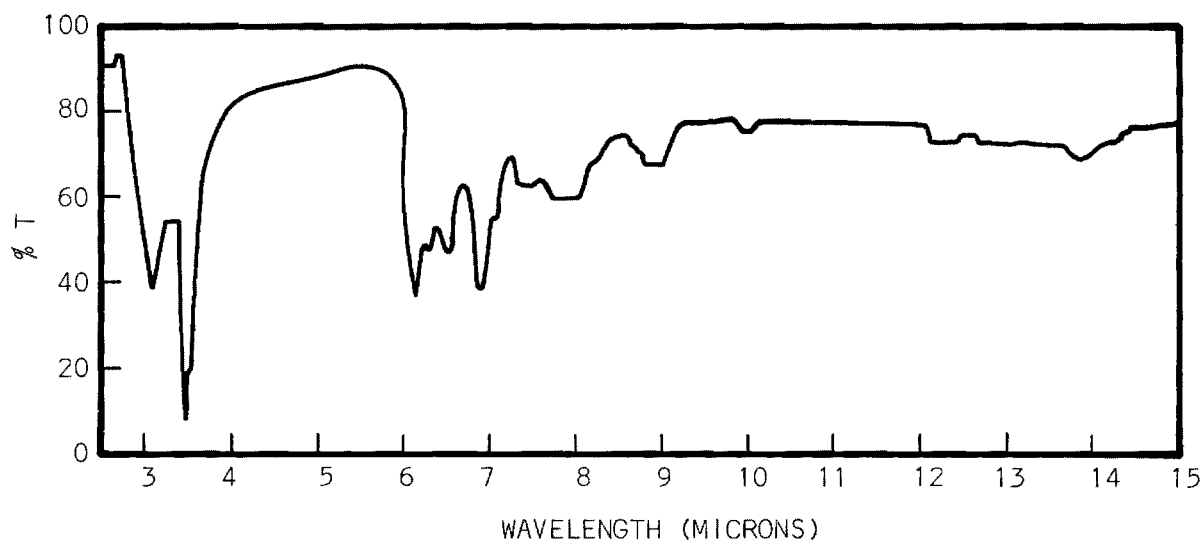


Figure 3. Dimerized Fatty Acid - Aliphatic Polyamine-type Resin Infrared Spectrum

GEORGIA INSTITUTE OF TECHNOLOGY

ENGINEERING EXPERIMENT STATION

ATLANTA, GEORGIA 30332

July 11, 1967

State Highway Department of Georgia
2 Capitol Square, S. W.
Atlanta, Georgia 30302

Attention: Mr. Leland S. Veal
State Highway Planning Engineer

Subject: Quarterly Progress Letter No. 10, Project A-802
"Hot Melt Traffic Marking Materials"
Contract No. 6401
Covering the Period from 1 April through 30 June 1967

Gentlemen:

Final observations of highway test stripes were conducted on June 14, 1967. Series I had received two years' weathering and Series II one year.

A large spread in performance was displayed within each series. Both contained some perfect stripes, and some severely failing stripes. As reported previously, priming is a major factor in developing maximum performance.

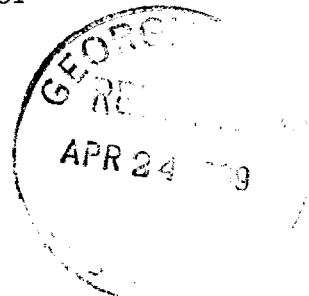
Details of the observations of these highway tests will be documented in the final report now in preparation.

Respectfully submitted,

W. R. Tooke, Jr.
Project Director

Approved:

for Frederick Baillinger, Chief
Chemical Sciences and Materials Division



GEORGIA INSTITUTE OF TECHNOLOGY

ENGINEERING EXPERIMENT STATION

ATLANTA, GEORGIA 30332

October 12, 1967

State Highway Department of Georgia
2 Capitol Square, S.W.
Atlanta, Georgia 30302

Attention: Mr. Leland S. Veal
State Highway Planning Engineer

Subject: Quarterly Progress Letter No. 11, Project A-802
"Hot Melt Traffic Marking Materials"
Contract No. 6401
Covering the Period from 1 July through 30 September 1967

Gentlemen:

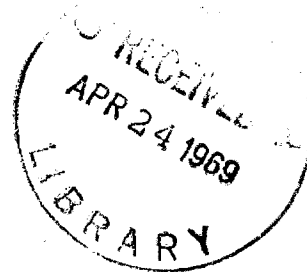
Project work during the current reporting period has been directed exclusively to the preparation of the Final Technical Report. The manuscript draft of this report is nearly complete, but several additional weeks' time may be required to complete figures and illustrations for the approval draft.

Respectfully submitted,

W. R. Tooke, Jr.
Project Director

Approved:

Frederick Bellinger, Chief
Chemical Sciences and Materials Division





GEORGIA INSTITUTE OF TECHNOLOGY
EXPERIMENT STATION

225 North Avenue, Northwest · Atlanta, Georgia 30332

May 22, 1968

State Highway Department of Georgia
2 Capitol Square, S.W.
Atlanta, Georgia 30302

Attention: Mr. Leland S. Veal
State Highway Planning Engineer

Subject: Quarterly Progress Letter No. 12, Project A-802
"Hot Melt Traffic Marking Materials"
Contract No. 6401
Covering the Period from 1 October through 31 December 1967

Gentlemen:

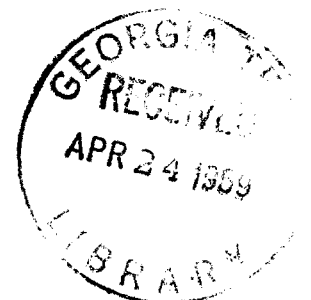
During this period all project work was directed to the completion of the draft of the Final Technical Report and preparation of included figures and illustrations.

Respectfully submitted,

W. R. Tooke, Jr.
Project Director

Approved:

Frederick Bellinger, Chief
Chemical Sciences and Materials Division





GEORGIA INSTITUTE OF TECHNOLOGY
EXPERIMENT STATION

225 North Avenue, Northwest · Atlanta, Georgia 30332

May 22, 1968

State Highway Department of Georgia
2 Capitol Square, S.W.
Atlanta, Georgia 30302

Attention: Mr. Leland S. Veal
State Highway Planning Engineer

Subject: Quarterly Progress Letter No. 13, Project A-802
"Hot Melt Traffic Marking Materials"
Contract No. 6401
Covering the Period from 1 January through 31 March 1968

Gentlemen:

During this period the draft of the Final Technical Report was completed and submitted to our Reproduction Service.

Respectfully submitted,

W. R. Tooke, Jr.
Project Director

Approved:

Frederick Bellinger, Chief
Chemical Sciences and Materials Division





EXPERIMENT STATION 225 North Avenue, Northwest · Atlanta, Georgia 30332

August 12, 1968

Mr. Leland S. Veal
State Highway Planning Engineer
State Highway Department of Georgia
2 Capitol Square, S.W.
Atlanta, Georgia 30334

Attention: Mr. Hugh L. Tyner, Sr.
Chief, Research & Development Branch

Subject: Quarterly Progress Report No. 14 covering the period from
April 1, 1968 through June 30, 1968.
"Hot Melt Traffic Marking Materials"
GHD Research Project No. 6401 Research Agency Project No. A-802
Project Director: W. R. Tooke, Jr.
Head, Industrial Products Branch
Research Agency: Georgia Institute of Technology
Engineering Experiment Station
Date Project Started: December 1, 1964
Date Project to be Completed: September 30, 1968

Gentlemen:

Project work during this reporting period has been as follows:

I - PROGRESS

(A) Project Work Plan Schedule

Project work is on schedule.

(B) Significant Technical Information

Editorial work responsive to the technical comments of Mr. B. Chaiken of BPR was initiated.

II - PROBLEMS

(A) Personnel

None



(B) Technical

None

III - FINANCES(A) Estimated Total Funds Expended:

	<u>During Quarter</u>	<u>Total to Date</u>
(1) Personnel	112.76	37,161.78
(2) Materials & Supplies	80.83	2,750.68
(3) Services	.00	.00
(4) Travel	.00	.00
(5) Equipment		
Rental	.00	.00
Purchase	.00	35.00
(6) Overhead (57%)	64.27	22,838.97
(7) Computer Services	.00	16.14

(B) Percent of Budget Expended: 97.76%(C) Percent of Contract Time Expended: 93.02%(D) Sufficiency of Funds

Sufficient

IV - OTHER PERTINENT INFORMATION(A) Project Personnel

None

(B) Significant Inspections or Correspondence Regarding Project

1. June 20, 1968 letter from L. S. Veal enclosed technical comments from Mr. B. Chaiken - BPR on Final Report Draft.

2. June 25, 1968 letter from L. S. Veal enclosed Supplemental Agreement No. 2.

August 12, 1968

(C) Technical Meetings and/or Presentations

None

(D) Recommendations or other Pertinent Information

None

V - EXPECTED DATE OF NEXT REPORT

(A) Annual Report

September 30, 1968

(B) Interim Report

Not Applicable

(C) Final Report

September 30, 1968

Respectfully submitted,

W. R. Tooke, Jr.
Head, Industrial Products Branch

APPROVED:

Frederick Bellinger

Chief, Chemical Sciences & Materials Division

Dup

FINAL TECHNICAL REPORT

PROJECT A-802

HOT MELT TRAFFIC MARKING MATERIALS

W. R. TOOKE, JR.

Research Contract No. 6401

October 21, 1968



Prepared for
State Highway Department of Georgia
in cooperation with
U. S. Department of Transportation
Federal Highway Administration
Bureau of Public Roads



Engineering Experiment Station
GEORGIA INSTITUTE OF TECHNOLOGY
Atlanta, Georgia

GEORGIA INSTITUTE OF TECHNOLOGY
Engineering Experiment Station
Atlanta, Georgia

FINAL TECHNICAL REPORT

Georgia Tech Project A-802

Research Contract No. 6401

HOT MELT TRAFFIC MARKING MATERIALS

by

W. R. Tooke, Jr.

October 21, 1968

Prepared for
STATE HIGHWAY DEPARTMENT OF GEORGIA
in cooperation with
U. S. DEPARTMENT OF TRANSPORTATION
FEDERAL HIGHWAY ADMINISTRATION
Bureau of Public Roads

The opinions, findings, and conclusions expressed
in this publication are those of the author and not
necessarily those of the State Highway Department
of Georgia or the Bureau of Public Roads.

FOREWORD

This project represented a logical follow-on of previous work under the same sponsorship directed at the development of test methods and formulations for traffic paints. The paint investigation entitled, "Use of Radioisotopes in Development of Test Methods and Formulations for Traffic Paints," Research Project HPS-1(60), was concluded in September 1965. The present study of hot melts has been conducted during the period December 1964 to December 1967.

The work has been performed within the Industrial Products Branch of the Chemical Sciences and Materials Division. Mr. W. R. Tooke, Jr., Branch Head, was Project Director. Other project personnel have included Mr. William Rose, Assistant Research Chemist--hot melt formulation work, Mr. David Hurst, Senior Research Technician--equipment design, materials application, and field evaluation, and Mr. Henry Horst, Student Research Assistant--laboratory testing. Dr. Harrison M. Wadsworth, Industrial Engineering has provided valuable assistance in experimental design and analysis, and Mr. Oliver Francis of the Rich Electronic Computer Center has programmed the analysis-of-variance computations. Other personnel of the Engineering Experiment Station have provided assistance as required. The Research Machine Shop, Design Services and Photographic Laboratory have been especially helpful.

The guidance and cooperation of engineers of the Georgia State Highway Department and of the U. S. Bureau of Public Roads is especially acknowledged, as well as the financial support of these agencies in sponsoring the project.

ABSTRACT

Extraordinary durability and quick-setting characteristics have firmly established hot thermoplastic as a preferred material for urban cross-walk delineation. However, its highway use for center striping has been hampered by high application costs and somewhat variable performance experience.

Laboratory and highway transverse-stripe testing has been performed on proprietary, specification-type, and experimental compositions of hot melts. The results of two years of transverse-stripe tests under very heavy traffic conditions have clearly confirmed earlier findings: hot melt service life is greatly superior to traffic paint.

The effectiveness of bonding agents for attaining durable adhesion to portland cement concrete was also demonstrated, conclusively. Concurrent laboratory researches on the field-tested materials and numerous variations provided indications of the interrelationships of composition, physical properties, application, and performance.

Hot melt traffic stripe material specifications were drafted based on physical property and laboratory performance test criteria. These specifications are believed to define the present state of the art and to provide suppliers with yardsticks for further developments.

Highway use of thermoplastic lines might remain highly selective as long as it represents merely a more durable replacement for traffic paints. Exploratory experiments have shown that thermoplastic lines can be textured so as to impart effective delineation under wet nighttime conditions.

A cost-effectiveness analysis of hot thermoplastic as compared with traffic paints indicated, at best, only marginal effectiveness for the hot thermoplastic. For most highway usage, development of the wet night visibility potential would be necessary to justify adoption of hot thermoplastic striping.

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I. INTRODUCTION

The concept of utilizing hot melt or thermoplastic striping compounds for traffic delineation dates back at least to World War II when the Road Research Laboratory in England explored various compositions of rosin, mineral oil pigments and fillers. Subsequently hot melts have become the dominant road marking medium in England. Similar broad acceptance in the United States has been inhibited by certain technical-economic factors which are discussed in several reports cited in the State of the Art Survey. However, numerous American municipalities are currently employing proprietary hot melts in traffic marking operations. New technical developments (including some of the findings of the present study) may lead to substantially increased usage of hot thermoplastic traffic marking media for highway marking as well as for urban streets.

The present and potential special merits of hot melts as compared with traffic paints include the following:

1. Maximum durability potential.
2. High visibility under all conditions.
3. Very rapid set (dry).
4. Cost-effectiveness advantages.
5. Unlimited package stability.

For these excellent reasons, interest in hot melt research and development has continued and further progress has been made. While the work of this project has been addressed mainly to solution of specific formulation problems,

objective evaluation of hot melt performance, and development of specifications; some attention has also been directed towards gaining an improved understanding of the physical requirements of the particular coating-substrate-environmental system.

II. OBJECTIVES AND WORK PLAN

The following objectives were originally proposed:

1. To make a comprehensive study and analysis of the prior art covering hot melt traffic marking materials and methods.
2. To evaluate existing formulation and application methods by accelerated and highway cross-stripe tests.
3. To develop such modifications in materials and methods as may appear necessary and desirable based on the findings of Objective 2.
4. To prepare specifications covering the best materials and methods developed under Objective 3.

Project work was sequenced generally in accordance with the objectives. An original plan to survey individual state highway departments for information on hot melt research was cancelled in view of the availability of an excellent current summary. In lieu of this, field observations were made of thermoplastic installations in progress and experienced engineering personnel were interviewed.

A need to place field tests so results could mature during the 3 year term of the project dictated that a major effort be made to attain workable formulations and experimental application equipment and methods as soon as possible. This activity dominated the first six months of the project period. Cross-stripe placements were made in June 1965.

Thereafter, attention was turned to formulation studies and physical property characterization of hot melts. The purpose was to seek hot melt improvements in accordance with Objective 3. This work was programmed toward a deadline of June 1966 for placement of a second series of "improved" hot melt cross-stripes. These placements were accomplished on schedule.

During the next six months additional attention was directed to thermoplastic resin blends in an effort to gain further formulation improvements, and work on specification test development was begun. By January 1967, attention was concentrated exclusively on the specification work and this was continued to the completion of experimental phases in June 1967.

Project activities were concluded with a final inspection of the test stripes in June 1967, subsequent detailed analyses of laboratory and field data, and the drafting of primer and hot melt specifications and this Final Technical Report.

III. STATE OF THE ART SURVEY

A combined literature and patent search was conducted with particular emphasis on locating recent patents covering hot melt traffic marking materials and equipment. Chemical Abstracts indices disclosed several new patents and publications of interest. The following items are cited as being particularly pertinent to the present study:

U. S. 3,036,928 (5/29/62) Poole, "Retroreflective Composition and Method of Applying Same."

This patent discloses hot melt compositions very similar to those investigated. The patent cites compositions based on non-drying alkyds as the primary resin. It was noted that the claims of this patent are very broad.

U. S. 3,020,252 (2/6/62) Hancock, "Road Marker Materials and Methods of Making Same."

Mixtures of sulfur, Thiokol and wood rosin are claimed in this patent.

Hancock, C. Kinney, I.E.C. 46, No. 11, pp. 2431-35, November, 1954.
"Plasticized Sulfur Compositions for Traffic Marking."

Discussion of details of developmental work on sulfur compositions is covered in this paper.

Swiss Patent No. 301,822 (12/1/54) "Plastic Mass for Road Marking."

This patent discloses hot melt compositions based on a rosin vehicle plasticized with "vaseline oil."

U. S. 3,018,704 Searight (1/30/62) "Stripe Applicator for Pavements."

This patent discloses an applicator which is currently in widespread commercial use.

Keese, C. J. and Fred J. Benson, Highway Research Board Bulletin 57, pp. 49-59 (1952) "Thermoplastic Striping Compounds."

This report covers experimental work by the Texas Engineering Experiment Station beginning in 1949. Formulation previously developed in England based on rosin-oil blends provided a point-of-departure for compositional studies. Slight modifications of the British formulations were found to yield a service life of better than three times that of painted stripes, and modifications of the binder with up to 15% of non-drying alkyd resin yielded further improvements. Adhesion to Portland cement concrete was never entirely satisfactory, however.

Reid, J. A., Barbara E. Sabey, and J. G. James, Journal of Applied Chemistry, 12, pp. 201- (May 1962), "White-Line Road-Markings and Light-Colored Road Surfacing."

In this survey of British practices much attention is given to thermoplastic markings. In England, thermoplastics have become the primary road-marking material. The following statement was noted, "The main conclusion drawn from all the road experiments is that hot-applied thermoplastic (normally 1/8 in. thick) is still the most durable and economic marking material for general use outside urban areas."

Much practical information is contained in a "Summary of Experimental Installations of Thermoplastic Pavement Striping Materials," dated April 13, 1964 and compiled by Mr. Bernard Chaiken of the Bureau of Public Roads Office of Research and Development. This report describes the performance of numerous

contract applications of proprietary hot melt compositions in various states. Evidence of good durability potentials was somewhat negated by variability of performance, high cost relative to paint, and specific problems of adhesion, bead retention, susceptibility to cracking, and snowplow damage.

In a report entitled "Plastic Marking Materials for Pavements" dated December 1964, the New York State Department of Public Works supplied much interesting detail on the modes of failure of thermoplastics. Despite certain shortcomings, they cite one installation of hot melt on asphalt that outlived six adjoining repaint applications and was still in excellent condition.

A number of other publications and patents of secondary interest have been reviewed.

Much valuable information was received through a personal visitation with Mr. Herbert A. Rooney, Senior Chemical Testing Engineer of the California Division of Highways. This information included copies of experimental compositional specifications for hot melt traffic marking material.

Finally, numerous manufacturers of hot melt materials, resins and other components have been most helpful in supplying technical data, formulation suggestions, and samples of materials. This cooperation was most essential in our endeavors to gain a current perspective on hot melt technology.

IV. MECHANICAL PROPERTY REQUIREMENTS

A. General

Hot melts differ from conventional traffic paints in such fundamental respects that the formulation technology is more nearly related to plastics than to conventional coatings. The common compositional features are, of course, the organic polymeric binder together with incorporated pigments and fillers.

In any case, the coating must be applied as a liquid which will rapidly convert to an adherent solid film capable of bearing traffic within minutes after application. A paint binder may be a solid or semi-solid resin dissolved in volatile solvent which evaporates from the applied film to achieve the re-conversion to solid. Obviously this mechanism is feasible only for thin films of perhaps 20 mils maximum thickness. Hot melts, on the other hand, are liquified by heat alone, applied hot, and re-convert to solid entirely by a cooling and "freezing" mechanism. Since no solvent escape is involved, films of 100-150 mils in thickness are entirely feasible. It is interesting to note that the application of paint by a "hot spray" technique has become increasingly popular, and represents a "wedding" of the two methods.

B. Melt Properties

An important limitation of hot melts is that melt viscosity (consistency) must not be so high as to retard the free flow of the liquified material in the application equipment. In practice, this means that the binder must be, predominately, a low molecular weight polymer with the physical property limitations that this inherently imposes.

From the viewpoint of polymer chemistry, this suggests that the binder resin should be of a type that can develop fairly strong intermolecular hydrogen bridges so as to minimize the disadvantages of low molecular weight. This type of bonding capability is also important for developing good adhesion. The physical chemical aspects of this subject have been discussed in some detail in a recent paper by Mark 1/. The practical consequences are that resins containing bridge-forming groups will be preferred for this application. Examples of such groups would include esters, ethers, amines, amides, hydroxyls, and carboxyls. At the same time, the bridge forming groups must not be so reactive as to introduce permanent cross-links and render the resin thermosetting. Further, the polymer must not degrade excessively at high temperatures, and when degradation occurs the products should not remain as carbonaceous residues.

The foregoing requirements are being attained in current practice by utilizing as the principal resin a rosin derivative and modifying this material by blending with a higher molecular weight polymer. Non-drying alkyds, cellulose ethers, acrylonitrile copolymers, and polyamides are among the modifying resins that are feasible. Undoubtedly other types of binder systems would be feasible providing the basic physical property limitations are observed.

Quantitative hot melt property requirements are detailed in subsequent sections of the report.

C. Applied Properties

Quite aside from the obvious dynamic stress factors of compression, shear, adhesion, flexure, abrasion, etc., as imparted by traffic; applied hot

melts must withstand stresses resulting entirely from differential thermal expansion and contraction against the substrate. The magnitudes of forces involved can be estimated from basic considerations.

D. Theoretical Stress Analysis

When a layer of thermoplastic is applied to Portland or bituminous concrete, either tensile or compressive axial stresses may occur in the plastic itself, and some shear stress may occur at the bonded interface between the plastic and concrete. The stresses might be considered to come from two sources: (a) cooling of the thermoplastic from its liquid state at 400° F to the range of environmental temperatures, (b) subsequent environmental heating and cooling of the bonded materials within a range of about 150° to 0° F.

At high temperatures, however, thermoplastics flow quite readily and thus stress-relieve themselves. Effect "a" and effect "b", in the higher temperature ranges, can therefore be considered negligible; the real range of interest in heating and cooling is probably from about 50° F downward, especially in the region where the plastic exhibits a relatively constant relation of stress to strain. This would be near and below the glass point of the material.

An expression for axial stress in the plastic due to thermal deformation may be derived from basic relations. If a strip of plastic and a strip of concrete are bonded together, the bonded strip will change in length with temperature. The individual strips, if unbonded, would also change in length with temperature, but by different amounts. Thus in a bonded strip each material will be deformed not only by temperature change but also by the other

material. The load necessary to cause deformation of one material by the other is transmitted through the bond. The total deformation of either material equals the sum of its thermal deformation and its deformation caused by action of the other material.

$$\delta_b = \delta_t + \delta_p \quad (1)$$

where δ_b = total deformation of the bonded strips, in.

δ_t = unrestrained thermal deformation of one material, in.

δ_p = the deformation of one material caused by thermal elongation (or contraction) of the other, in.

The total deformation, δ_b , of the plastic and concrete strips, when bonded, is the same, thus,

$$\delta_b = \delta_{t1} + \delta_{p1} = \delta_{t2} + \delta_{p2} \quad (1a)$$

where subscripts 1 and 2 refer to plastic and concrete respectively.

The expression for thermal deformation is:

$$\delta_t = \alpha L(T - T_o) \quad (2)$$

where α = thermal coefficient of expansion (or contraction) in/in °C.

T = temperature °C

L = length, in.

The expression for load deformation (strain) of a material is:

$$\delta_p = \frac{PL}{AE} \text{ (Hooke's Law)} \quad (3)$$

where P = load, lb.

A = cross-sectional area, sq. in.

E = modulus of elasticity, psi.

Substituting equation (2) and (3) in equation (1a) yields:

$$\alpha_1 L(T - T_o) + \frac{PL}{A_1 E_1} = \alpha_2 L(T - T_o) + \frac{PL}{A_2 E_2} \quad (4)$$

In practice, a plastic strip will be applied to a concrete pavement, which will deform very little by action of the plastic. The last item of equation (4) is therefore negligible, and,

$$\alpha_1 L(T - T_o) + \frac{PL}{A_1 E_1} = \alpha_2 L(T - T_o) \quad (4a)$$

or

$$s = \frac{P}{A} = (\alpha_2 - \alpha_1) (T - T_o) E \quad (5)$$

where s = axial stress in plastic, psi.

subscript 1 has been dropped for E and A of the plastic, since these terms for concrete are no longer in the equation.

The sign of "s" in equation (5) follows the convention that tensile stresses are positive and compressive stresses negative.

The temperature T_o is one at which stress relief has been obtained by cold flow of plastic. This would normally be somewhat above the glass point of the plastic, at the upper limit of the temperature range in which the plastic has a fairly well defined modulus of elasticity. The thermoplastic materials, however, creep even at low temperatures; thus T_o might be close to the ambient temperature during a protracted period of cold weather. Because of this characteristic, the most severe condition is anticipated to be a hard, sudden freeze, rather than a long cold spell.

Above a temperature of about 50° F, computed stresses have little meaning, since at such temperatures the plastics flow quickly enough to relieve them before they can build up. If T_0 has fallen to a low value during cold weather, a sudden warming could conceivably cause compressive stresses. Compressive stresses probably will not occur otherwise, since compression takes place under warming conditions.

A graphical illustration of stress variation with temperature is shown as Figure 1. Values have been estimated (from handbook references) for E and α . A plot based on experimental data might be somewhat different. Tension is indicated by a plus sign and compression by minus.

These computations indicate that realistic temperature changes can induce tensile or compressive loadings that may easily exceed the ultimate stress of the hot melt material. Moreover, once failure is initiated in tension or compression (a crack develops) then high shear loads are imparted to the substrate bond in the area of failure, and the destructive effect is aggravated by the resulting loss of adhesion.

Theoretical aspects of the mechanical effects of highway traffic on paint films have been discussed in a previous report 2/. Hot melt coatings may be expected to be subject to effects similar to paints with two exceptions. (1) The higher profile of the hot melt may be expected to increase shear-impact loadings, and, (2) the greater thickness of hot melts would require consideration of shear and compressive as well as tensile moduli in deriving a theory of mechanical failure. A quantitative theoretical treatment was beyond the scope of the present project. Rather, we have undertaken to measure mechanical properties of experimental formulations, and relate these properties to observations of service performance.

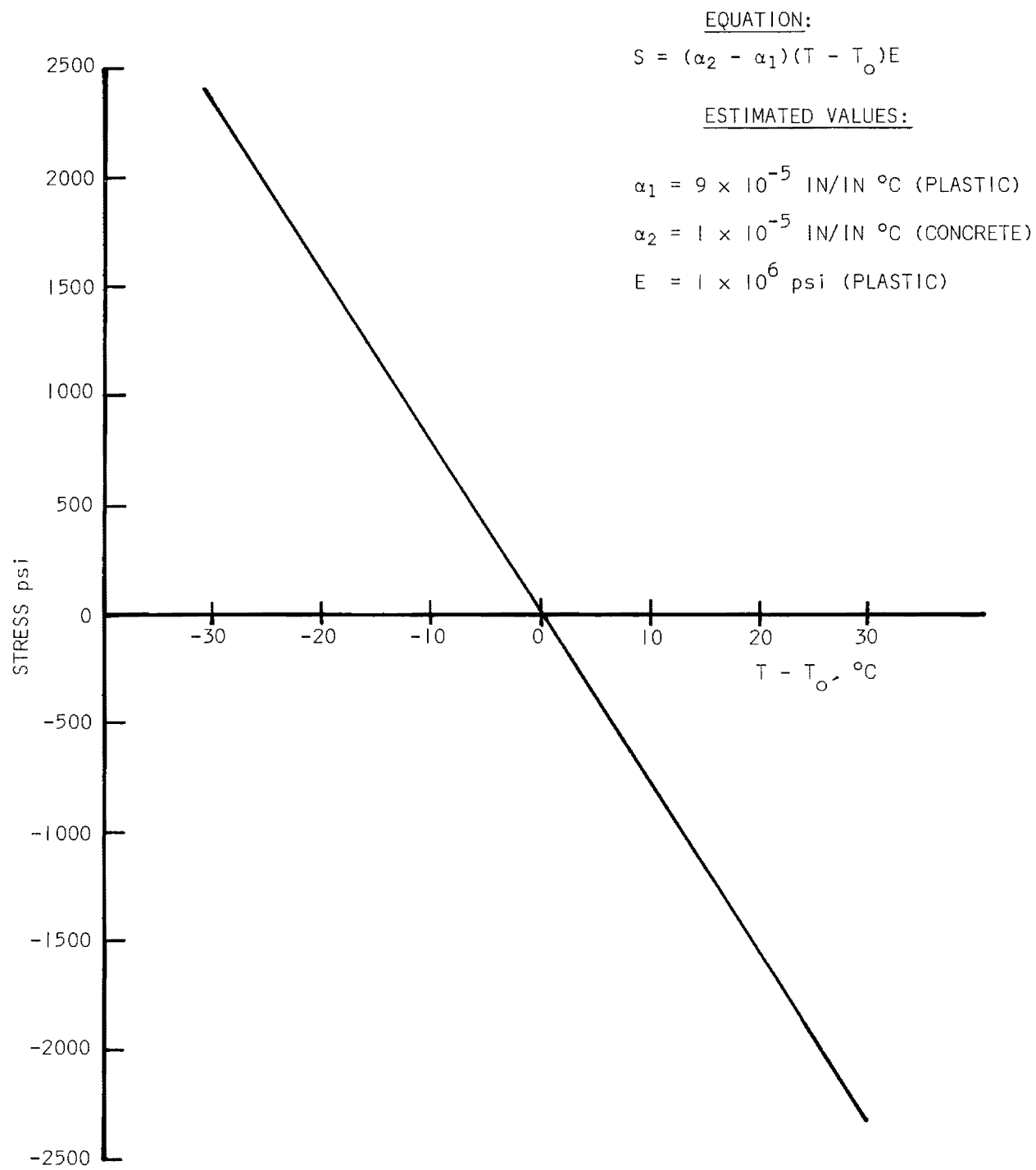


Figure 1. Stresses in applied thermoplastics caused by temperature changes.

V. EXPLORATORY PREPARATION AND CHARACTERIZATION

A. Proximate Analysis

Much information on formulations was available from the "State of the Art Survey." Of all such material, a compositional specification entitled "Experimental White Thermoplastic Traffic Line Formula 1R353" by the State of California Division of Highways was judged to be most representative of a current field-tested known composition. A copy of this specification is included in Appendix B. A sample quantity of this material was prepared in accordance with the specification. Quantities of two proprietary thermoplastic line materials were also obtained for examination. The purpose of the proximate analysis work was to establish a point-of-departure for formulation studies on the basis of materials of known performance.

Specimens of each material were dispersed in hot toluene, and the filler content was separated by centrifugation, dried and weighed. Resin content was estimated by difference. The composite filler was screened to remove fine pigment, and the coarse residue was digested in HCl. This dissolved all granular material (apparently calcium carbonate), leaving glass beads as the final residue.

The results of these analyses are shown in Table I together with determinations of approximate bead size, of phthalic anhydride in the resins and of density. The findings delineated the general area of beading and filler loading practice and suggested the probable use of alkyd resins in the binder component of proprietary hot melts.

TABLE I
HOT MELT PROXIMATE ANALYSIS

Material	Composition by Weight, Per Cent		
	Proprietary A	Proprietary B	Calif. Spec.
Glass Beads	35	21	21
Pigment, excluding beads	40	64	54
Resins	<u>25</u>	<u>15</u>	<u>25</u>
Total Weight Per Cent	100	100	100
	Other Data		
Approximate bead size, mils	15	10	15
Phthalic acid, qualitative	present	present	absent
Density, lbs/gal	18.0	17.3	16.5

B. Laboratory Preparations and Properties

1. Materials and Formulation Approach

During the exploratory period, major effort was being directed toward attaining as rapidly as possible a set of hot melt formulations for placement on highway transverse stripe tests as Series I. The formulations of Series I were not intended specifically as "advances" in the state-of-the-art, but rather to provide field application experience, performance feed-back and guidance for subsequent systematic studies. We were interested in surveying several basic types of binder resins as the compositional variable in this study. Throughout this report, reference will be made to numbered formulations which are detailed as to composition in Appendix A, Table XVIII and as to physical properties in Table XIX. The formulations identified with this exploratory work would include Nos. 1 through 173. The binder materials of primary interest were styrene-acrylate resin, ethyl cellulose, petroleum derived resins and rosin derivatives.

2. Preparation and Laboratory Testing Methods

Suitable heavy-duty mixing equipment is required to disperse properly even small batches of hot thermoplastic. For our preparations we adapted a variable-speed drill press with a heavy-duty sweep paddle impeller fitted to a stainless steel beaker. The beaker rests on a hot plate and is restrained by a girth-clamp. The assembly is shown in Figure 1 together with spare beaker-paddle units. The unit is made with a 4000 cc beaker and can handle a batch up to 6000 g.

A batch is normally prepared by wetting and blending the composite binder under low speed continuous agitation until a uniform melt is attained.

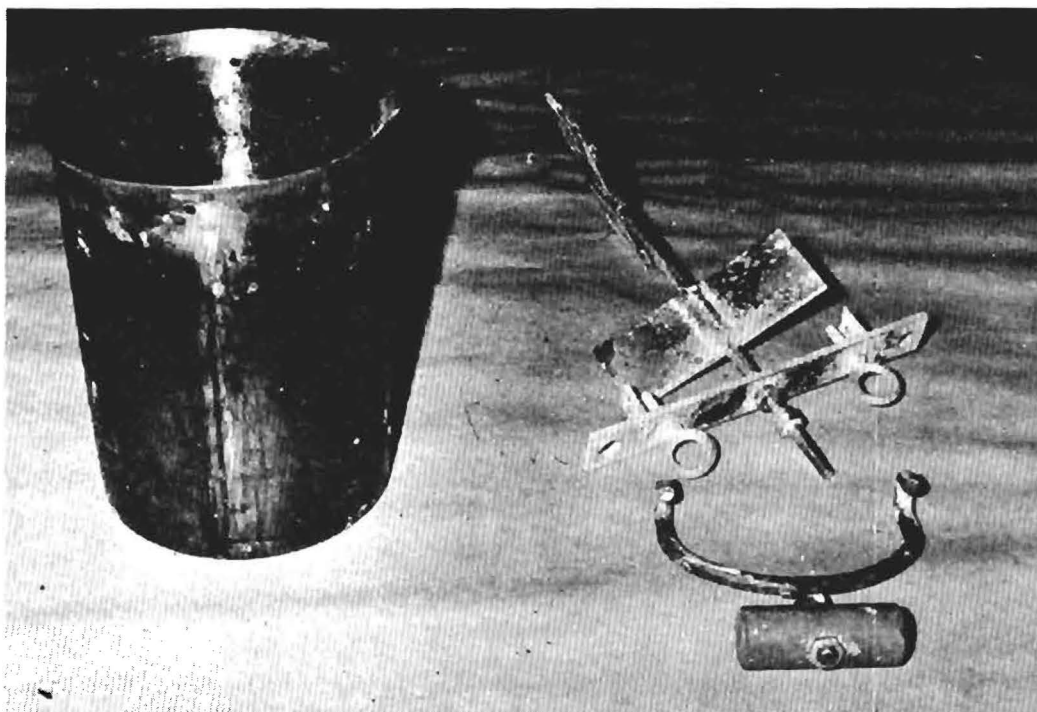
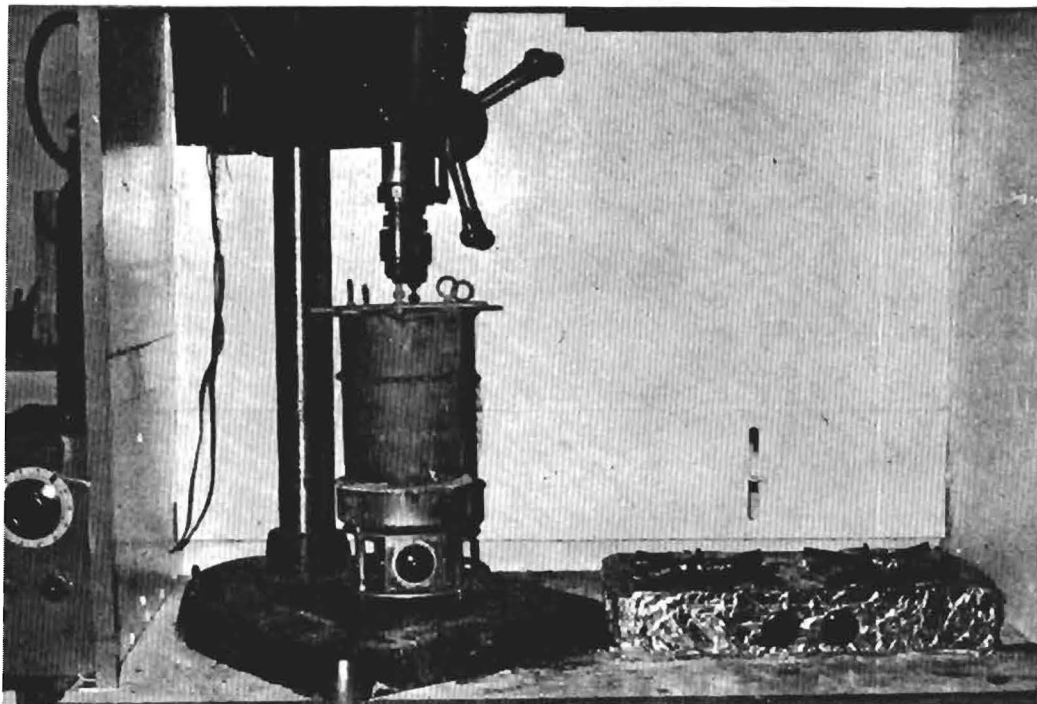


Figure 2. Mixing equipment.

At this point, a small specimen can be withdrawn and cooled to check compatibility. During the melting operation, the melt temperature is maintained below about 350° F. Filler materials are next introduced gradually, titanium dioxide first and coarser materials last. Agitation speed and temperature may be raised during this period to improve dispersion action. Mixing is continued for at least ten minutes after final filler addition. The molten batch is then poured off into shallow pans and allowed to cool and solidify into slabs.

The formulations comprising Series I were evolved largely on the basis of subjective comparisons of consistency, application, adhesion and flexibility. However, laboratory testing methods for the purpose of formulation guidance were developed during the exploratory period. These methods are described in detail in Appendix B. Interpretations of the purpose and significance of the various routine laboratory tests will be discussed in Section VI. SYSTEMATIC FORMULATION STUDIES.

Certain other test methods were explored, and later abandoned for routine use. Methods and data of potential usefulness are summarized as follows:

Thermal Coefficient of Expansion

A device was constructed to measure the coefficient of linear thermal expansion of hot melts in accordance with ASTM D-696-44.

Experimental data on selected hot melts are given in Table II. Difficulties were experienced in obtaining precision values of the thermal coefficient. It was concluded that this property would not vary sufficiently in response to formulation variations to provide useful formulation guidance.

TABLE II
COEFFICIENTS OF LINEAR EXPANSION

Sample Size: 0.5 x 0.5 x 8.0 in.		
Temp. Range: 0 - 70° F		
Sample		Coefficient of Expansion
HM No.	Item	
108	Proprietary B	2.5×10^{-5}
64	Proprietary C	2.8×10^{-5}
26	California Spec.	2.5×10^{-5}
27	Ethyl Cellulose	2.3×10^{-5}
	Concrete	1.1×10^{-5}

Tensile Test

Initially we planned to make routine determinations of tensile properties of plastics in accordance with ASTM D 638-64T. One set of test specimens was prepared and run on an Instron machine equipped with extensometers. The data is given in Table III. The preparation of satisfactory test specimens was found to be difficult and time consuming and the testing requires rather painstaking manual operations. Accordingly, the tensile test was abandoned as a routine method, since other testing techniques were available that could provide adequate stress-strain characterization.

C. Development of Primers

Prior experience by others (literature survey) has disclosed that hot thermoplastics tend to exhibit poor adhesion to P/C concrete. To correct this shortcoming, two types of bonding primer have been used in service applications.

The first of these is a nitrile rubber/phenolic resin blend supplied in volatile solvents at a concentration of about 10%. This material is commercially available to comply with detailed compositional specifications under the trade name Pliogrip AD 574, supplied by Goodyear Chemicals Division^{*}. A quantity of this product was obtained for test work. It is ready to use as received and requires no catalyst.

The second type of primer is a clear catalyzed epoxy material. Since this product was available only as a proprietary material, formulation research was undertaken to develop satisfactory compositional specifications. The primer that was developed consisted of a polyamide-epoxy "alloy" which cures in place under the heating influence of the overlaid hot thermoplastic.

^{*} This vendor also furnishes a very similar product designated PlioBond 20. The two products differ only in solids content.

TABLE III
INSTRON TENSILE TEST

Gage Length: 2 inches

Width: 0.5 inches

Thickness: 0.75 inches

Cross-sectional area 0.375 square inches

Sample: Proprietary A #109

Sample	Max Stress (psi)	Elongation at Failure (%)	Load Rate (in/min)
1	426	9.47	0.2
2	493	1.80	1.0
3	548	4.50	0.5
4	533	9.00	0.5
5	705	8.00	0.5
6	600	15.00	0.5
7	547	8.75	0.5
8	600	18.50	0.5
9	505	6.25	0.5
10	440	17.50	0.5

Mean Stress at Failure 538.4 psi

Compositional specifications which include both types of primers are given in Appendix B. Laboratory data indicating the bond improvement attained with these primers are shown in Table IV. With four distinct types of hot melts, shear bond strength is greatly improved by the primers when the bonds are tested both at room temperature and under sub-freezing conditions. The constituents of the primers were specified generically and optional sources of all materials were established as available.

The performance of these primers was subsequently investigated in the highway cross-stripe testing program, and those results (reported in detail later in this report) fully confirmed the laboratory findings.

The effects of moisture in concrete on bond strengths was also investigated in connection with the primer development work. Laboratory shear bond test assemblies were prepared from dry concrete blocks (moisture content 4%) and from damp concrete blocks (moisture content 7.5%). The damp blocks did not exhibit any visible surface wetness. Test results are presented in Table V. Clearly, damp concrete causes a total loss of bonding of hot thermoplastic to the unprimed surface. By use of primers, most of the bond loss is restored. Thus, in addition to improving dry bonding strength, primers provide a "margin of safety" to attain adequate bonds where highway surfaces may be slightly (if not visibly) damp.

TABLE IV
PRIMER EFFECTS - BONDING TO DRY CONCRETE

Test Method: Shear Bond Test								
Replications: 5								
Hot Melts								
No.:	109		27		28		26	
Type:	Proprietary A		Ethyl Cellulose		Alkyd		Calif. Spec.	
Temperature:	<u>0° F</u>	<u>75° F</u>	<u>0° F</u>	<u>75° F</u>	<u>0° F</u>	<u>75° F</u>	<u>0° F</u>	<u>75° F</u>
(Maximum Shear Stress, psi)								
No Primer	45	395	1	45	8	135	3	290
Rubber/Phenolic	475	484	450	490	95	170	355	490
Epoxy	481	292	400	406	125	268	420	360

TABLE V
SUBSTRATE MOISTURE AND PRIMING EFFECTS ON BONDING

Test Method:	Shear Bond Test	
Replications:	5	
Test Temperature:	75° F	
Hot Thermoplastic:	Proprietary A, #109	
<u>Priming Treatment</u>	<u>Concrete Condition</u>	
	<u>Dry (4.0 moist.)</u>	<u>Damp (7.5% moist.)</u>
	<u>Maximum Shear Stress, psi</u>	
No Primer	395	0
Rubber/Phenolic	484	243
Epoxy	292	247

VI. SYSTEMATIC FORMULATION STUDIES

A. Filler Properties

The fillers in hot thermoplastic marking material have several functional purposes. The first and most important is to reduce the cost of the product by replacing some of the volume of the resinous matrix material with a lower cost solid. This objective was investigated in some detail. Crystalline calcium carbonate (dry ground marble) was ideally suited for this purpose, since it is low in cost, hard, durable, white, and available in a wide range of particle sizes.

Another function of fillers is to provide continued retro-reflective properties of the line material throughout its service life. For this purpose, glass beads are incorporated in the matrix so that as the plastic material wears down, new beads may be exposed at the surface. This theory was accepted as valid in the present investigation, and was not subjected to controlled tests. The very slow abrasive wear of the hot thermoplastic as observed in service tests, did suggest that much of the incorporated beading may never be exposed. Significant economy could be realized if the incorporated glass beads could be replaced with the granular filler.

Finally, a whitening or coloring pigment is required to impart the desired appearance. Rutile titanium dioxide has been used throughout the present study to achieve whiteness. While this is the most economical "whitening" pigment available, it is, nonetheless, an expensive ingredient in the hot melt composition. Accordingly, experimental work to determine a optimum TiO_2 loading is economically important.

Theoretically, a type of fine fibrous filler could be of interest as a means of reinforcing the mechanical properties of hot melts. Some preliminary work was done along these lines utilizing asbestos fines. This work was abandoned after a few experiments disclosed that necessary flow properties in hot melts are lost before sufficient fiber can be introduced to improve significantly the mechanical strength.

1. Filler Packing Density

a. Particle packing theory. By computing optimum distributions of particle sizes, the void volume of granular material may be very substantially reduced, with a corresponding increase in apparent density. The void volume parameter is utilized effectively in some very successful schemes of concrete mix design, and it finds use in paint technology in the form of the critical pigment volume concentration (CPVC). In hot melts, the use of void volume ideas offers the potential for reducing significantly the cost of compositions, while improving application and performance characteristics. The particle packing theories are discussed in considerable detail by Dalla Valle 3/. The practical objective is to attain by computation or experiment a distribution of particle sizes such that increasingly finer particles can most completely occupy all void spaces between coarser particles. Computational approaches to the problem are based on assumptions of spherical particles and, therefore, can provide only general guidance for practical work. In practice, systematic experimental determinations of packing densities are always required.

b. Packing density measurement. In hot melt compositions of special interest the filler constituent has the following composition:

	<u>Wt. %</u>
Titanium Dioxide	17.2
Ground Marble (RO-40)	55.2
Glass Beads (coarse)	27.6

The titanium dioxide in this filler is of such fine particle size ($0.5\ \mu$) that it may be regarded appropriately as a part of the resin matrix, and thereby ignored as a filler constituent. Attention may thus be concentrated on the mixture of ground marble and glass beads. These relatively coarse materials are also more amenable to the measurement method as outlined below:

Packing Density Measurement Method

Selected quantities of the filler components are individually weighed and combined to yield about 400 grams of composite filler. The composite filler is violently agitated on a paint shaker in a one quart can for five minutes, and then poured into a 500 cc graduated cylinder. The cylinder bottom is then tapped (bounced) uniformly for 25 strokes on a rubber mat to attain maximum consolidation. Final volume is observed and apparent density (weight/volume) computed.

c. Densities of composite fillers. The first study of packing densities was undertaken on the premise that higher density might be attained by incorporating some fine glass beads in the mixture. The apparent density response surface for the system Ground Marble (RO-40) - Coarse Beads-Fine Beads is shown in Figure 3 on a ternary plot of percentage composition by weight with the apparent density parameter presented in the form of contour

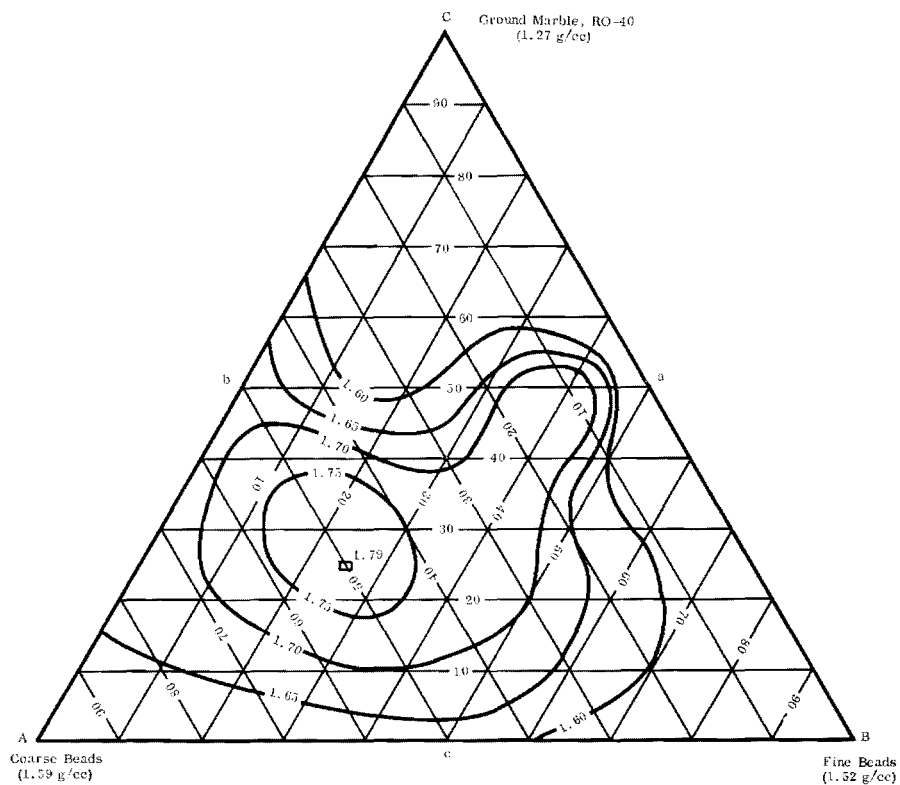


Figure 3. Apparent density of the system ground marble, RO-40 - coarse beads - fine beads.

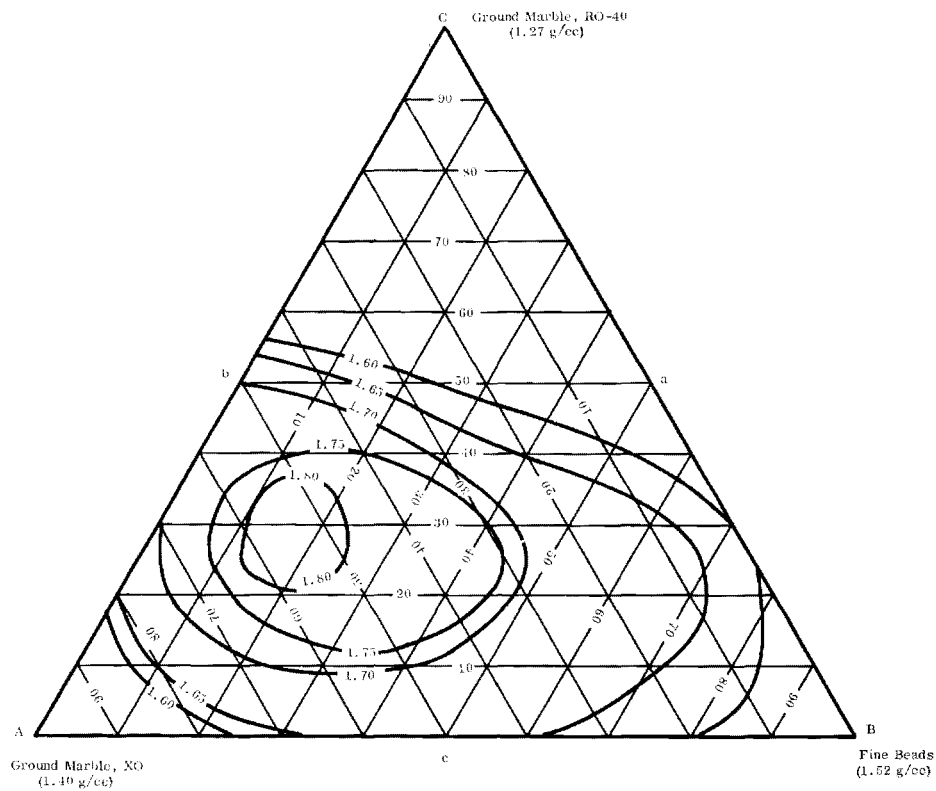


Figure 4. Apparent density of the system ground marble, RO-40 - ground marble, XO - fine beads.

lines. In this investigation it was discovered that at the maximum density point, only 25% of ground marble is combined with a total of 75% of coarse and fine beads. This was believed to be an excessive bead concentration. Furthermore, the use of fine rather than coarse beads to obtain maximum retro-reflection is indicated in the work of Pocock and Rhodes 4/. Therefore, the use of a coarse ground marble to replace the coarse beads was proposed.

A suitable coarse ground marble designated as Ground Marble, XO was obtained, and another ternary study was completed on the system Ground Marble, XO-Ground Marble, RO-40-Fine Beads as shown in Figure 4. This time maximum density was obtained in a compositional area of about 20% of fine glass beads, up to 40% might be used without serious loss of packing efficiency.

The general significance of these findings is that optimum packing densities may be readily determined and should provide very helpful guidance in selecting filler compositions.

d. Compounding effects. The filler composition represented by the maximum density point in Figure 3 was substituted in a control (California Specification - Hot Melt No. 1) on a weight replacement basis. The temperature-viscosity curve of the resulting Hot Melt No. 25 was then determined. (The Consistency Determination procedure is described in Appendix B.) The result is shown in Figure 5 as compared with the control. Note the significantly lower viscosity of the new composition. This appears to be a clear demonstration of the reduction of the "binder demand" of a hot melt formulation by the adjustment of filler size distribution to obtain minimum voids.

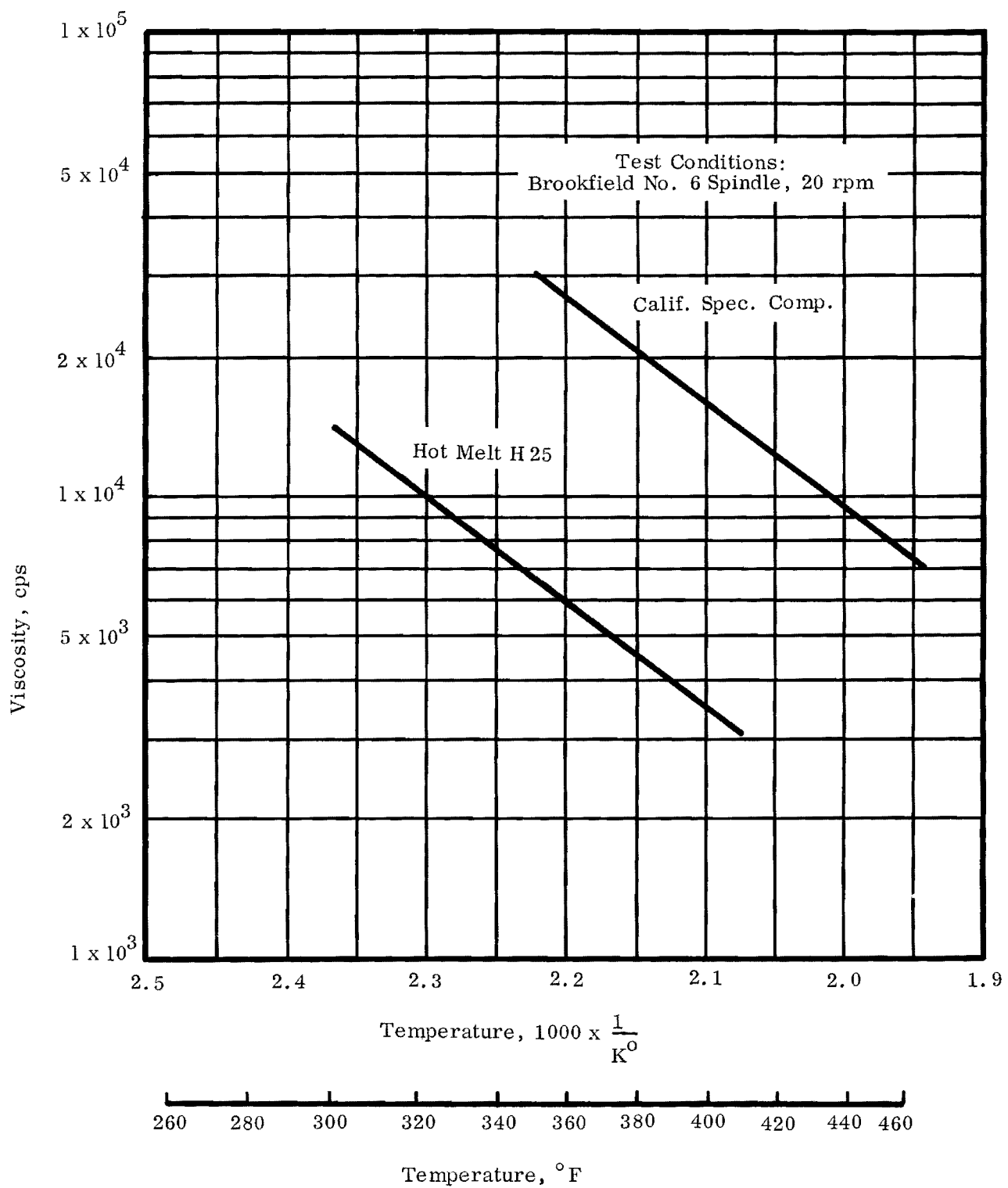


Figure 5. Effect of filler substitutions on viscosity.

2. Titanium Dioxide Pigmentation

Levels of TiO_2 pigmentation between 0% and the 13% utilized in the California Specification (#26) were explored experimentally. Opacity and whiteness were judged to be adequate at a TiO_2 level of 3.5%. Vehicle color has been observed to affect whiteness significantly; therefore, we have concluded that TiO_2 requirements should be based on appearance properties rather than an arbitrary compositional percentage. This can permit more economical utilization of this relatively costly ingredient.

B. Vehicle Properties

1. Consistency

The consistency or flow properties of hot melts have been referred to repeatedly from both theoretical and empirical viewpoints. This most important property is primarily a vehicle function. Experimental determinations of vehicle consistencies have demonstrated that the unfilled resin melts exhibit a consistency of approximately one log less than the corresponding filled composition (75% filler). This trend is shown in Figure 6. The knowledge of required vehicle consistency is of material assistance in exploratory work with novel resins.

2. Compatibility

Coatings chemists are generally justified in the view that a cloudy vehicle will exhibit poor film properties. More tolerance of cases of borderline incompatibility was found to be appropriate with hot melts. Often, some haziness in the molten resin blend and in the cooled mass would,

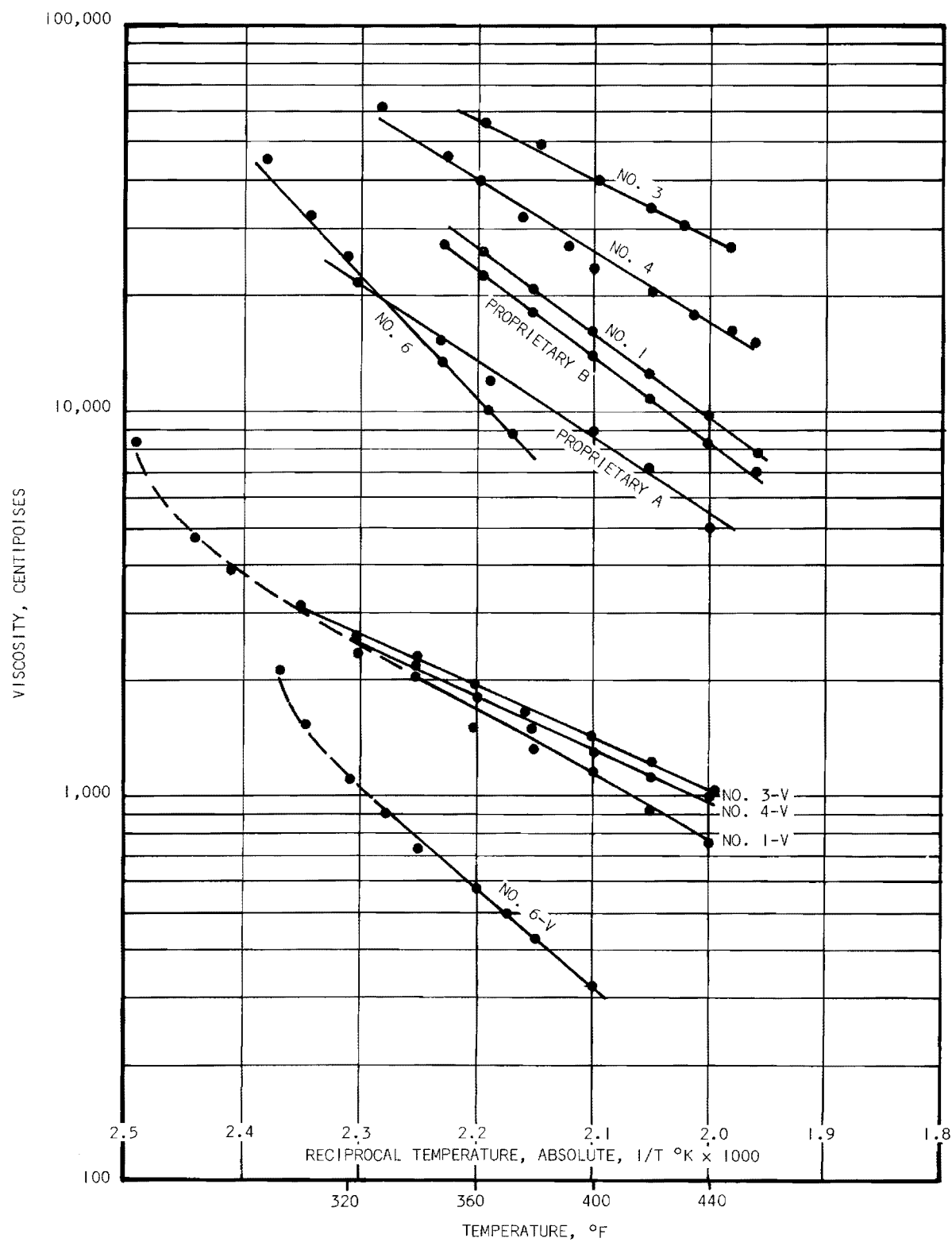


Figure 6. Temperature - viscosity relations.

nevertheless, yield very satisfactory properties in the filled composition. This was particularly true where efforts were made to introduce high polymers to enhance physical properties.

On the other hand, certain resin blends appeared to be quite homogeneous in the "clear," but when combined with fillers, these systems would tend to separate a clear supernatant vehicle phase whenever agitation ceased. Such compositions were unusable.

In summary, it was not feasible either to accept or reject a particular resin blend on the sole basis of compatibility observations.

3. Mechanical Tests

The possibility of specific interactions of fillers with vehicle resins tended to limit the practical usefulness of quantitative mechanical property characterization of "clear" solid resin blends for the purposes of the present investigation. Direct comparative data on two resin systems, filled and unfilled are shown in Table VI. (The several test methods are discussed in the next section - VI.C. Composite Properties.) These systems are seen to exhibit rather uniform changes in properties upon filling. If a research program were more narrowly focused at details of vehicle formulation, then an even more comprehensive characterization of hot melt vehicle properties should be rewarding.

In one sense, we did utilize mechanical property characterizations of vehicle resin blends quite extensively in developmental formulation work. Specimens of blended resin were carefully examined manually--probed, flexed, and handled. Subjective comparisons provided much guidance in adjusting the proportions of vehicle components to attain a desired combination of properties.

TABLE VI

MECHANICAL PROPERTIES OF THERMOPLASTIC VEHICLES

<u>Formulation Type</u>	<u>Creep Rate, in/in/min</u>	<u>Flexural Impact @ 77° F</u>		<u>Consistency @ 350° F, cps</u>
		<u>Max. Fiber Stress, psi</u>	<u>Absorbed Energy in-lbs/in²</u>	
Alkyd:				
Unfilled (#46)	20	226	1.25	320
Filled (#65)	6	860	1.08	26,000
Ethyl Cellulose:				
Unfilled (#59)	15	2,380	3.3	-
Filled (#60)	5.5	4,550	4.2	12,000

While this activity cannot be reported in explicit detail, most of the composite formulations which have been studied did evolve from initial subjective characterizations.

C. Composite Properties

1. Planning and Test Selection

The basic purpose of the laboratory formulation of hot thermoplastics was to gain information about the potential usefulness of various raw materials (particularly resins and modifiers) and, if possible, to develop compositions of improved economy and performance.

At the outset, our field performance information was limited to the knowledge that "acceptable" performance had been attained with several known compositions and proprietary products in service applications. Full field testing of any novel composition was expected to require several years. Thus, to accomplish any meaningful formulation work, it was expedient that laboratory testing be utilized to assess relevant properties of experimental formulations as compared with "known" products. Unfortunately, the relevance of a given property or test could not be evaluated directly against quantitative field data. Inferences from the "known" products were of some help, but primary guidance was derived from a "common sense" assessment of the property requirements of service conditions. In the light of our earlier discussion of mechanical property requirements, perhaps we are justified in describing this "common sense" as the application of principles of engineering mechanics to a conceptual model of the performance situation for thermoplastic traffic lines.

Essentially, we recognized that the service failure of thermoplastic lines would arise from imposed stresses that would eventually cause excessive flow, rupture, loss of adhesion, abrasive erosion, or combinations of these failure modes. Thus, the substrate bond and the stress-strain properties should adequately characterize performance potential. Undoubtedly, stresses in tension, compression, flexure, and shear occur, and loading rates vary from very slow (thermal effects) to very fast (traffic). A viscoelastic thermoplastic material will be expected to undergo creep in response to slow loading rates, and to become increasingly elastic and even brittle at very high rates. At low temperatures (0° F) elastic-brittle response may be anticipated even for low loading rates.

From these considerations a group of formulation developmental tests were selected. Details of the methods are described in Appendix B. Some interpretive comments follow.

Consistency measurements were used to establish approximate upper and lower limits on the flow properties of hot melts. However, within these limits other viscoelastic properties affect the application quality of a formulation. This fact was not observed until the time of application of Highway Series II--after most of the laboratory formulation and testing was completed. In particular, we found that polyamides tend to contribute a large elastic component to the flow response. This is detrimental to the application qualities even though the measured consistency may be near the lower limits. We were unable to measure this elastic component with our viscometer, and could detect it with certainty only by test applications. More research is needed on practical rheological characterization of hot melts.

Aside from the foregoing limitations, it was found that consistencies of less than about 2000 cps are undesirable because the molten material flows excessively on the concrete and does not retain a uniform edge and design thickness. On the other hand, at viscosities in excess of 35,000 cps, the slow flow retards the application rate prohibitively. The operator tends to "get ahead" of the flow and "starve" the extruder gate. This also causes uneven edges, varying thickness, and a rough top surface of the applied line. Undoubtedly adhesion is also affected adversely by high consistencies.

Low consistency can usually be corrected by reducing the operating temperature, but high consistency is not similarly correctable. Excessive resin degradation would occur if temperatures were increased beyond the standard level of about 400-450° F in the melting pot.

Flexural Creep Properties were determined from experimental load-deflection curves. Flexure testing was selected because it was, experimentally, the most convenient method to use both for creep and impact loading. Standard test moldings in the form of long rods of 0.5 x 0.5 in. cross-section were easily prepared uniformly and in quantity. Creep test conditions were selected somewhat arbitrarily to permit a test to be completed in a few minutes time. Load-deflection creep curves were plotted routinely, but most of the data has been reported as an empirical Creep Rate. This parameter together with Elastic Deformation and Modulus are defined precisely in the test method description in Appendix B. Creep testing was confined to room temperature (77° F), since in preliminary experiments extremely low creep rates were exhibited for all practical materials at subfreezing temperatures.

Obviously, the creep rate is a parameter of consistency or flow of thermoplastic at room temperatures. If a thermoplastic exhibited excessive creep it might be expected to smear and spread on the road. If creep were very low, however, the thermoplastic might be too rigid to relieve accumulated stresses, and cracking or disbonding might be promoted. Guidance as to appropriate creep rates for best performance must be derived from field experience. The direct formulation significance of elastic deformation and modulus was somewhat obscure. After a number of runs we abandoned making these measurements. The elastic modulus in tension-compression would be a basic parameter in computing stresses arising from temperature changes and stress distributions occurring under impact loadings.

Static Shear Bond Strength tests served the important purpose of evaluating adhesion of thermoplastics to a concrete substrate. Priming was not used in this test, since one purpose of the formulation studies was to attain improved adhesion to bare concrete.

Flexural Impact Tests were intended to measure the strength and toughness of thermoplastics under loading conditions approximating that of traffic. Maximum Fiber Stress is the impact strength parameter, and absorbed energy is the toughness parameter. Tests were performed at room temperature (77° F) and at low temperature (0° F) to assess the temperature effect.

2. Summary of Findings

a. Effects of additives to a standard formulation

The purpose of this study was to evaluate in the alkyd-type formulation the individual effects on physical properties of various vehicle

additives. Each additive was incorporated by substitution at a level of 10% by weight of the original composite vehicle. This substitution treatment included the original components of the formulation as well as other resins. Formulation details are given in Table VII. Test results are detailed in Appendix A, Table XXIII, Formulation Nos. 36-52.

On the basis of their physical property effects the additives were classified as hardening, plasticizing, reinforcing, or extending. This classification is presented in Table VIII. Some materials were placed in two categories, since they exhibited dual effects. Two materials, sulfur and polyurethane elastomer were ruled out for practical use because of emanations of objectionable and toxic vapors from the melts. Epoxy resin was highly detrimental to shear bond strength, although it greatly enhanced flexural impact properties. Among the reinforcing resins, special interest was directed to ethyl cellulose and polyamides.

b. Further compositional searches

A large number of hot melt formulations were prepared and tested in a broad effort to find means of attaining over-all property improvements and/or to reduce materials costs. This work encompasses Formulations 53-95. The prior systematic study of additives provided the basis for much of this work.

Evaluation of numerous formulation variations based on ethyl cellulose and on half-second cellulose acetate butyrate further confirmed the ability of cellulosics to impart toughness to hot melts. At the same time, the thermal stability of the cellulosics was found to be very marginal. Severe discoloration often occurred.

TABLE VII
HOT MELT ALKYD MODIFICATIONS

<u>Basis Formulation</u>		<u>% by Wt.</u>
Alkyd (VBR-5010)		10
Stabilite #10		10
Pentalyn K		5
Small Glass Beads		25
Marble, RO-40		21.5
Marble, XO		25
TiO ₂ -R-610		<u>3.5</u>
		100
<u>Additives</u>		
(Replacing 10% of Composite Vehicle in Basis Formulation)		
<u>Hot Melt No.</u>		
36	Pentalyn K	
37	Stabilite #10	
38	Alkyd (VBR-5010)	
40	Estane (Polyurethane Rubber)	
41	Maleic Modified Ester (VBR-201)	
43	Eastbond M-5 (Amorphous Polypropylene)	
44	Ethylcellulose	
45	Epon 1007	
46	Pliolite AC	
47	Abitol	
49	Polyamide (Versamid 900)	
50	SAIB (Eastman Chemical Company)	
51	Sulfur (Stauffer)	
52	Polycarbonate (Lexan - General Electric)	

TABLE VIII
CLASSIFICATION OF RESINS BY FUNCTION

HM No.		Creep Rate (in/in/min $\times 10^{-3}$)	Flexural Impact (77° F)		General Characteristics
			Max. Fiber Stress (psi)	Absorbed Energy (in-lb/in ²)	
	<u>Hardening</u>				
41	Maleic Resin	6	488	1.0	Low creep Moderate to high impact energy
44	Ethyl Cellulose	10	1000	1.36	
45	Epoxy	4	1401	2.04	
46	S/A Resin	6	860	1.08	
52	Polycarbonate	2	1095	1.10	
	<u>Plastizing</u>				
38	Alkyd	600	89	0.82	High creep Moderate impact energy
47	Hydrogenated Rosin				
	Ester	100	1095	1.30	
40	Polyurethane	600	956	2.43	
	<u>Reinforcing</u>				
40	Polyurethane	600	956	2.43	Variable creep High impact energy
43	Polypropylene Gum	70	796	1.67	
49	Polyamide	29	1000	1.34	
44	Ethyl Cellulose	10	1000	1.36	
45	Epoxy	4	1401	2.04	
52	Polycarbonate	2	1095	1.30	
	<u>Extending</u>				
50	SAIB	50	860	1.03	Little change in physical properties
37	Hydrogenated				
	Ester Gum	50	410	0.78	
43	Polypropylene Gum	70	796	1.67	

Various efforts to upgrade the over all physical properties of alkyd-based formulations appeared to be only partially successful. Subsequent findings from field testing indicated, however, that the performance potentials of alkyd types may be considerably better than physical property data would suggest. Clearly, these materials remained strong candidates for use in hot melts.

Other work was directed at variations on the theme of the "California Spec." in which the S/A resin was replaced partially or completely with epoxy, polycarbonate, polyester, cellulosics, or combinations thereof. None of the results appeared especially encouraging, but the demonstrated variety of compatible combinations was interesting.

c. Polyesters and polyamides

An elastomeric polyester was found to exhibit very high impact properties when utilized in unextended form, but all efforts to extend this resin were unsuccessful. Better results were obtained with a "hard" polyester. Extremely high impact properties were attainable with appropriate plasticizer incorporation (Formulation No. 136), and outstanding bond strengths were observed. A tendency for high consistencies was characteristic of the polyester. Also, it would not accept large volumes of extender resin without prohibitive losses of physical properties. In view of these limitations, attention was concentrated on polyamides.

The toughness and bond strength attainable from polyamide formulations at feasible consistencies was most striking. A wide variety of plasticizing and extending resins (See Formulation Nos. 132-1 through 132-13) were found to be compatible, and to confer various combinations of properties. Interest

finally centered on the use of polyamide resin plasticized with O, P,-toluene ethyl sulfonamide (Santicizer No.8) and extended with hydrogenated rosin triglyceride. Three formulations of this general type were prominent among the items included in Highway Series II.

VII. HIGHWAY CROSS-STRIPE TESTING

A. General Considerations

At the project planning stages, it was recognized that field testing would necessarily involve a large amount of scaling-down from "normal" stripe placement procedures. A study of commercial hot melt placement equipment indicated no basic difficulty in achieving placements of small quantities of material substantially identical with commercial placements. Details of the equipment developed are described below. Close observations of placements with several types of commercial equipment disclosed no evidence of application differences as compared with the experimental unit.

A scheme of cross-stripe or transverse stripe placement on the highway was selected as opposed to longitudinal placement for the following reasons:

- (1) This procedure has been standardized for testing of traffic paints under ASTM A 713-46.
- (2) Close comparisons of formulation variations may be studied on a homogeneous substrate.
- (3) No evidence has been documented to indicate a lack of correlation of transverse placements with longitudinal placements.
- (4) Both economic and technical conclusions rest on relative performance of hot thermoplastics as compared with traffic paint rather than on absolute measures of service life.
- (5) The scheduling of the project required that any feasible acceleration of wear effects on the highway tests be utilized to attain significant performance distinctions in the testing time available.

B. Equipment

Commercially available equipment for hot melt application is designed for practical service use. The smallest units available, with a capacity of 150 pounds, were still much too large for the scale of operations that were anticipated in this program.

The critical problem was that a quantity of less than 30 pounds was to be placed at one time, and the equipment was then to be purged rapidly for placement of the next formulation. As many as eight different formulations were to be placed in several hours' time. The melting time delay precluded the possibility of using a single combined melter-applicator. The problem was resolved by constructing a simple multiple batch premelter, and a small easily cleaned self-heated applicator.

1. Premelter

This unit was designed to service the field applicator with up to four separate batches of pre-melted material. It consisted essentially of a four place gas stove shielded on all sides and serviced with gas from a 20 L. P. gas bottle. Batches were melted in 5-gallon pails.

The premelter required continuous attention of one man to bring the batches to working temperature, and to maintain the desired temperature until each batch was transferred to the applicator. The standard working temperature was 400° F. No difficulties were experienced with the premelter operation.

2. Field Applicator

The requirements of small size and ease of cleanout for quick batch turnover were accomplished by close-coupling a two gallon capacity box reservoir to a scaled-down extruder foot assembly. These elements were mounted on a four wheel dolly together with a beader unit, and with gas infra-red heaters for both the reservoir and the extruder. The unit is shown in Figure 7. After making minor modifications and gaining operating experience, we were able to maintain very satisfactory hot melt temperature control in the range of 400-450° F, and to apply uniform lines with excellent thickness control.

It was necessary for the operator to develop some manipulative skill, and to have assistance constantly at hand during field applications. A minimum team for the work consisted of the operator, mechanic-assistant, re-supply man, and premelter attendant.

C. Highway Tests, Series I

1. Purpose

A fundamental question essential to the effective pursuit of laboratory development work was "What are the relationships of composition, physical properties, and performance?" Cost-effectiveness economics was also of importance, even in this first series, and this was related to secondary questions of substrate effects, priming treatments and thickness of application.

Thus the broad purpose of the Series I tests was to become acquainted with field application techniques, and hopefully, to obtain answers to



Figure 7. Experimental hot-melt stripe applicator.

numerous technical questions. These answers should provide guidance for formulation and for subsequent test series.

2. Experimental Plan

Eight formulations were selected for use in Series I. These included two proprietaries, three variations of the California Specification, and three formulations embodying other resins.

The conventional application thickness of hot melts is a nominal 1/8 in. for this program, thicknesses of 3/32 in. and 3/16 in. were chosen to evaluate the thickness effect.

Portland cement (P/C) concrete was known to yield generally poorer and more erratic performance than asphaltic concrete as a substrate for hot melts. Applications were to be made to both paving types.

Additionally two types of priming treatments, an epoxy and a rubber-phenolic resin solution, were to be evaluated for effectiveness in improvement of hot melt adhesion to P/C concrete.

The foregoing independent variables were to be examined in factorial experiments. Two types of painted stripes were to be applied simultaneously or performance comparisons. As in all previous highway testing, the paints were to be applied at nominal 10, 15, and 20 unit thicknesses. All applications were representative of typical painting conditions. No surface preparations were employed.

The entire experimental design of Highway Series I is summarized in Table IX. We should emphasize that Factorial Experiments A, B, and C were drawn from a common pool of test stripes which contained only a single replication of a given treatment combination. This was done to facilitate analysis of variance with a computer program designed for full factorial experiments.

TABLE IX
DESIGN OF SERIES I
FACTORIAL EXPERIMENTS

		Factorial Experiment			
		A	B	C	D
Materials:					
Proprietary A	(109)	X	X	X	
Proprietary B	(108)	X	X		
Cal. Spec.	(15)	X	X		
1/2 S/A	(16)	X	X		
2 S/A	(19)	X	X		
H. C.	(21)	X	X		
Ethyl Cellulose	(24)	X	X		
Alkyd	(22)	X	X		
Alkyd Paint	(104)				X
Chlor. Rub.-Alkyd Paint	(105)				X
Thickness:					
3/32 in.		X	X	X	
3/16 in.		X	X	X	
10 mils					X
15 mils					X
20 mils					X
Priming:					
None		X	X	X	
Rubber-Phenolic		X		X	
Epoxy		X		X	
Substrate:					
Concrete		X	X	X	X
Asphalt			X	X	X
Observed Wear Periods:					
12 mos.		X	X	X	X
24 mos.		X			

3. Placements and Observations

Applications of Highway Series I were made on Interstate 85 at Lenox Road Bridge on June 1-3, 1965. The site location is shown in Figure 8, and photographs of the application work are in Figure 9. The surface was clean and dry. Cross-stripes were placed in the outer north-bound lane on P/C concrete immediately south of the bridge and on asphaltic concrete immediately north of the bridge. Priming of P/C concrete was done with a paint roller about 5 minutes ahead of stripe application. A total of 80 stripes were placed, numbered 301-354 on P/C concrete and 355-380 on asphalt. The thickness of all stripes was measured and photographs were taken immediately after placement. A subjective rating of "application properties" was also recorded on a 1-10 scale.

Observations and photographs were made at quarterly intervals and data was recorded on IBM card formats. Subjective observations of stripe integrity were recorded utilizing ASTM D913-51 (chipping), ASTM D 661-44 (cracking), and ASTM D821-47 (abrasion, erosion) photographic standards. Night visibility was measured with the Hunter Night Visibility Meter in accordance with ASTM D1011-52 (night visibility). Observations were limited to those areas of the stripes which are subjected to heaviest tire action; the extremes and the center (between treads) were not considered.

The stripes on asphalt were unexpectedly covered with repaving after only 12 months observations. The stripes on P/C concrete were observed for a full 24 months as originally intended.

4. Data Analysis and Discussion

The wear of the stripes as a group was sufficiently slow that the observations at 24 months (12 months on asphalt) provided satisfactory characterizations of relative performance. While all raw observations have

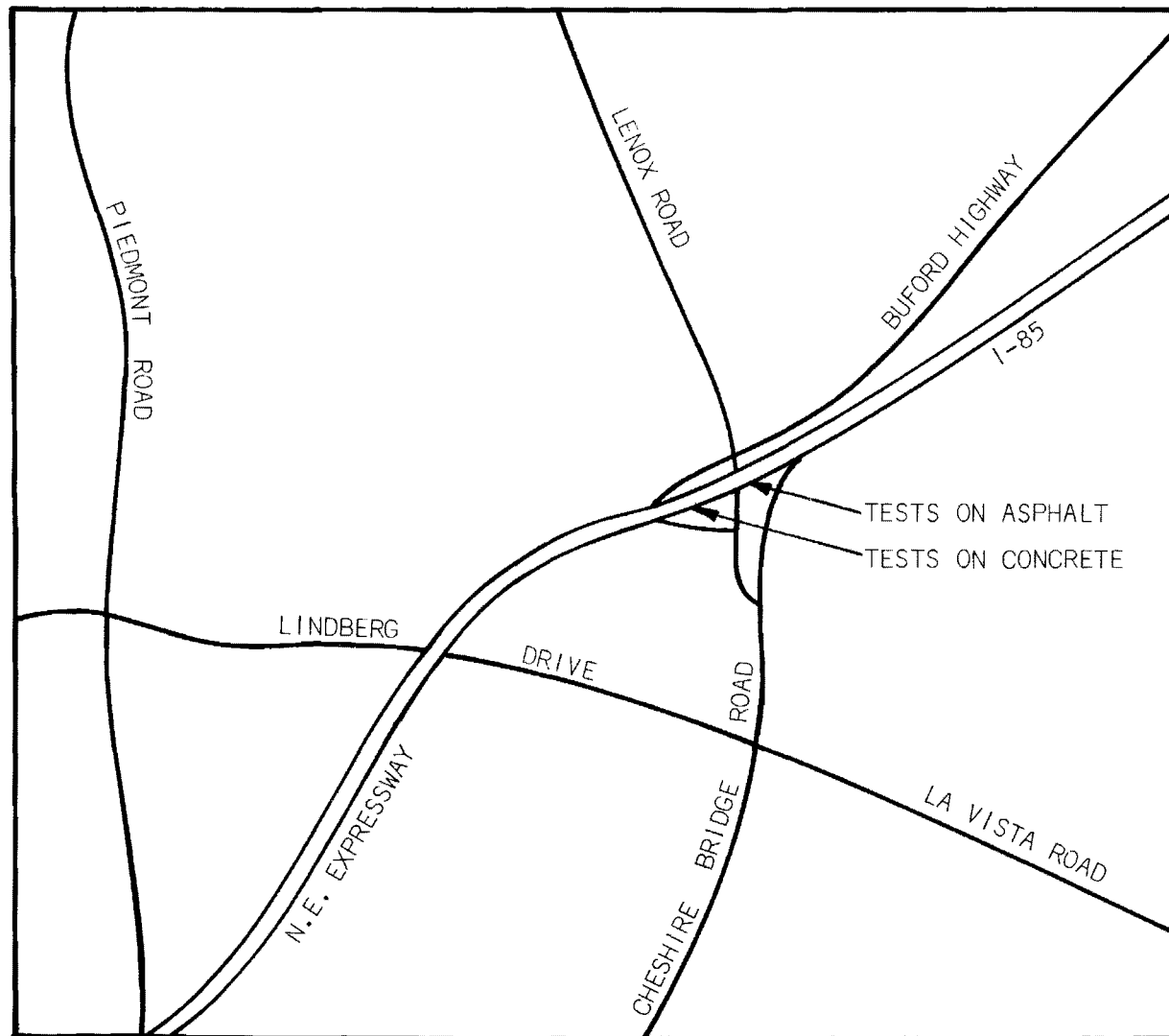


Figure 8. Location of highway tests.



Figure 9. Photographs of highway application work.

not been included as such in this report, those observations which pertained to each analysis-of-variance were tabulated by the computer program in a systematic presentation of individual treatment combinations and mean values. These appear in Appendix A immediately following the F-ratio table for each analysis.

The results of all of the variance analyses have been consolidated into two tabular presentations. Table X shows average values and 95% confidence limits for Integrity and single line observations of Night Visibility on P/C concrete only at 12 and 24 months service. Table XI shows the substrate effect (P/C concrete vs. asphalt) where priming is excluded. This data was available only at the 12 months period. Appropriate data on the control paints were inserted in the tables for comparison; the paint data were not subjected to analysis of variance routines.

The integrity data at 12 months of Factorial Experiment C is shown in Table XII. This was a small experiment to determine if priming might be needed on asphalt substrates. The conclusions appeared to be evident without further analysis.

The pertinent findings of the foregoing data analyses are summarized as follows:

Substrates

The loss of all test applications on asphalt after only 12 months wear considerably impaired our determination of substrate effects. The available data indicates that hot melts perform slightly better on asphalt than on unprimed concrete, but our experiment did not provide statistically significant proof of this effect. Table XII shows a much larger effect of the substrate for this one material than does the over-all average.

TABLE X

HOT MELT PERFORMANCE AVERAGES ON P/C CONCRETE ONLY

Hot Thermoplastic No. Description		12 Months			24 Months		
		Integrity *		Night Visibility **	Integrity *		Night Visibility **
		\bar{X}	95%		\bar{X}	95%	
			C.L.			C.L.	
			($\pm ts$) \bar{X}			($\pm ts$) \bar{X}	
108	Proprietary B	7.0	0.7	10.0	5.2	0.9	9
109	Proprietary A	7.0	0.7	8.0	7.0	0.9	6
16	$\frac{1}{2}$ S/A	8.2	0.7	11.0	7.8	0.9	5
15	1 S/A	7.5	0.7	12.0	6.3	0.9	4
19	2x S/A	4.7	0.7	5.0	4.2	0.9	2
21	Hydrocarbon	7.5	0.7	7.0	5.7	0.9	3
24	Ethyl Cellulose	8.2	0.7	10.0	7.3	0.9	11
22	Alkyd	8.3	0.7	9.0	7.8	0.9	5
104	Reg. Alkyd Paint	3.0	-	5.3	1.3	-	1
105	Chlr. Rubber Paint	2.3	-	2.6	1.3	-	1
Thickness:							
	3/32"	6.8	0.3		5.9	0.5	
	3/16"	7.7	0.3		7.0	0.5	
Priming:							
	None	6.5	0.4		5.2	0.6	
	Rubber	6.8	0.4		6.2	0.6	
	Epoxy	8.5	0.4		7.7	0.6	

* Primed and unprimed

** Observations on one line only

TABLE XI

HOT THERMOPLASTIC PERFORMANCES AVERAGES ON P/C CONCRETE AND ASPHALT WITH NO PRIMING
AT TWELVE MONTHS ONLY

Hot Thermoplastic No. Description		12 Months, Concrete and Asphalt				12 Months, Concrete Only				12 Months, Asphalt Only			
		Integrity*		Night Visibility		Integrity*		Night Visibility		Integrity*		Night Visibility	
		95%		95%		95%		95%		95%		95%	
		C.L.		C.L.		C.L.		C.L.		C.L.		C.L.	
		\bar{X}	($\pm ts$)	\bar{X}	($\pm ts$)	\bar{X}	($\pm ts$)	\bar{X}	($\pm ts$)	\bar{X}	($\pm ts$)	\bar{X}	($\pm ts$)
108	Proprietary B	7.0	2.1	10.8	1.6	5.5	1.5	10.0		8.5	4.3	11.0	
109	Proprietary A	6.7	2.1	6.5	1.6	7.0	1.5	8.0		6.5	4.3	5.0	
16	$\frac{1}{2}$ S/A	7.5	2.1	11.4	1.6	7.5	1.5	11.0		7.5	4.3	12.0	
15	1 S/A	6.3	2.1	11.0	1.6	6.0	1.5	12.0		6.5	4.3	10.0	
19	2x S/A	4.3	2.1	4.5	1.6	4.0	1.5	5.0		5.0	4.3	4.0	
21	Hydrocarbon	6.3	2.1	6.5	1.6	7.5	1.5	7.0		5.0	4.3	6.0	
24	Ethyl Cellulose	6.5	2.1	10.0	1.6	6.5	1.5	10.0		6.5	4.3	10.0	
22	Alkyd	7.7	2.1	8.5	1.6	8.5	1.5	9.0		7.0	4.3	8.0	
104	Reg. Alkyd Paint	3.8	-	4.5	-	3.0	-	5.3		4.6	-	3.7	
105	Chlr. Rubber Paint	3.3	-	2.2	-	2.3	-	2.6		4.3	-	2.0	
Thickness:													
	3/32"	6.1	1.0			5.87	0.6			6.87	2.2		
	3/16"	7.0	1.0			7.25	0.6			6.87	2.2		
Substrate:													
	P/C Concrete	6.6	1.0	9.0	0.8								
	Asphalt	6.9	1.0	8.3	0.8								
Thickness x Substrate:													
	P/C Concrete 3/32	5.9	0.6										
	3/16	7.2	0.6										
	Asphalt 3/32	6.9	0.6										
	3/16	6.9	0.6										

* Unprimed only

TABLE XII
INTEGRITY DATA FOR PROPRIETARY HOT
THERMOPLASTIC B AT TWELVE MONTHS

	Substrate			
	P/C Concrete		Asphalt	
Film Thickness:	3/32"	3/16"	3/32"	3/16"
Priming:				
None	4	7	8	9
Rubber	5	8	8	8
Epoxy	9	9	10	9

Another consideration is that some formulations exhibited a greater tendency to crack over the asphalt substrate. This cracking was treated as an integrity failure even though it was not accompanied by any actual loss of material by chipping. Also, some of the cracks were caused by cracks in the asphalt substrate. These facts, we believe, justify the conclusion that hot melts attain superior adhesion to asphalt than to unprimed concrete. Further, we believe that priming of asphalt will not significantly enhance stripe performance, and that the performance on unprimed asphalt will approach that attained on primed concrete.

Thickness

Superior performance was obtained from the heavy film, 3/16", in all experiments. While the effect was not conclusive on asphalt, it was significant at the 1% probability level on concrete.

The application operation was also better with the 3/16" film. The molten plastic flowed more uniformly and rapidly under the gate in the applicator shoe, and a higher speed of application could be maintained. The feasibility of service applications of films of 3/32" or less with existing types of equipment is considered to be very doubtful.

Priming

We have dismissed priming of asphalt as contributing nothing significant to stripe performance. In distinct contrast, the effect of priming P/C concrete is very highly significant. The rubber-phenolic primer provides some improvement but epoxy primer greatly enhances stripe integrity.

Formulations

Assessment of relative integrity performance of the various formulations on asphalt is distinctly inhibited by the limited wear period of only 12 months. No statistically significant data were obtained. In most cases the performance ordering of formulations on asphalt appeared to be similar to that observed on concrete.

Data on stripe integrity of various formulations on concrete yielded very significant results. A summary plot of this information is shown in Figure 10. Mean values of integrity are centered in horizontal bars representing the 95% confidence limits of the mean. The X's represent the individual treatment combination yielding the highest integrity value. In all cases this was the 3/16" thick epoxy-primed stripe. Finally, Night Visibility and Application Ratings are tabulated at the right margin.

First, it is noteworthy that no single formulation is outstandingly superior in all respects, but all hot melts are far superior to the control paints. An examination of the integrity performance curves of the various materials as shown in Figure 11 indicates that the service life (time of wear to integrity = 5) of an average of all the hot melt stripes on concrete could be 7 or 8 times longer than paint. Since many individual stripes are at the 9-10 level of integrity at 24 months, one may conclude only that their potential service life must be many times that of paint.

With the exception of Formulations #19 and #21, the laboratory prepared materials compared favorably with the proprietary hot melts. Compositions of very satisfactory performance could be built around special rosin esters modified with appropriate quantities of styrene-acrylate, ethyl cellulose, or non-drying alkyd resins.

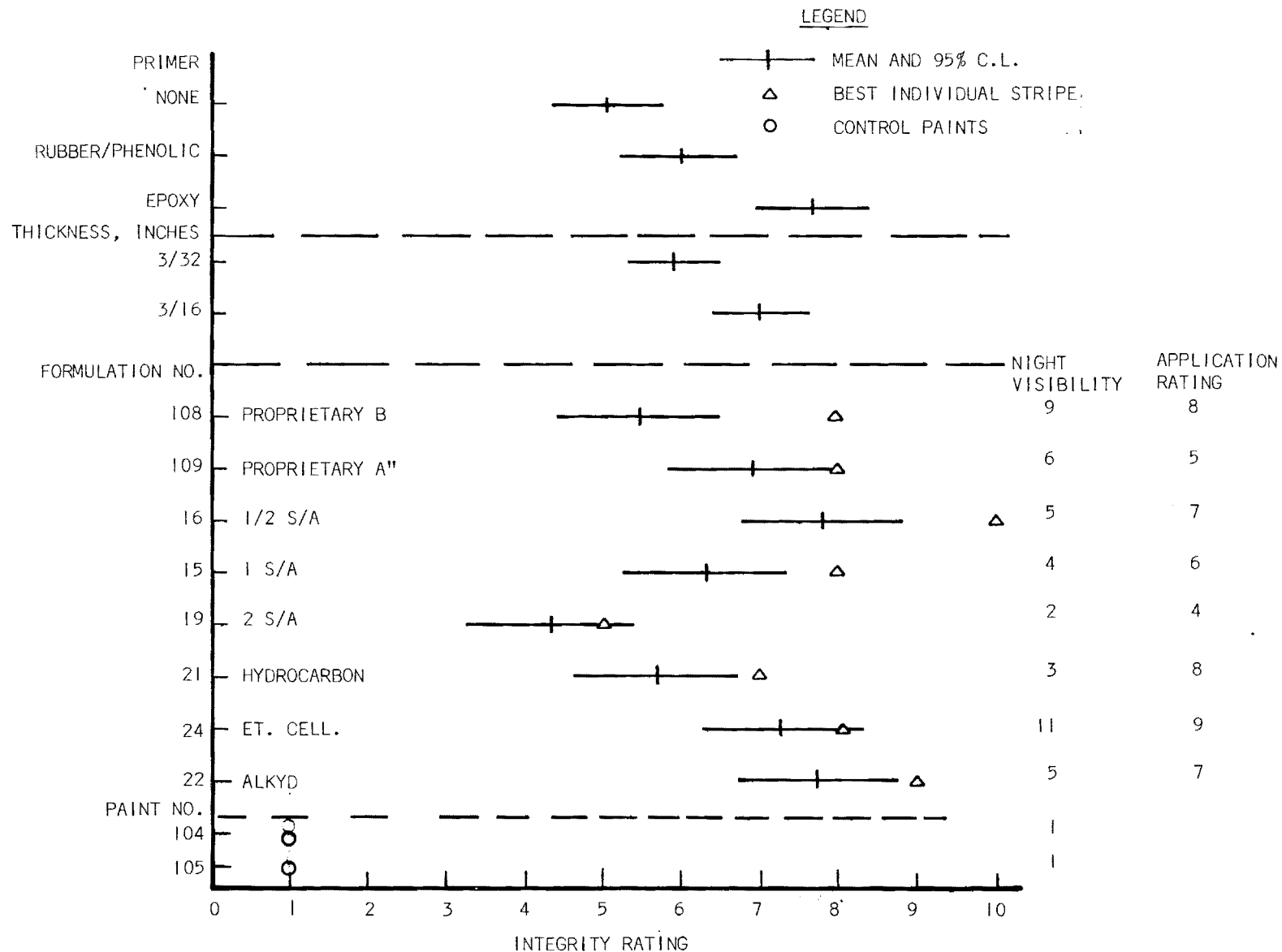


Figure 10. Summary of performance at 24 months on concrete, series I.

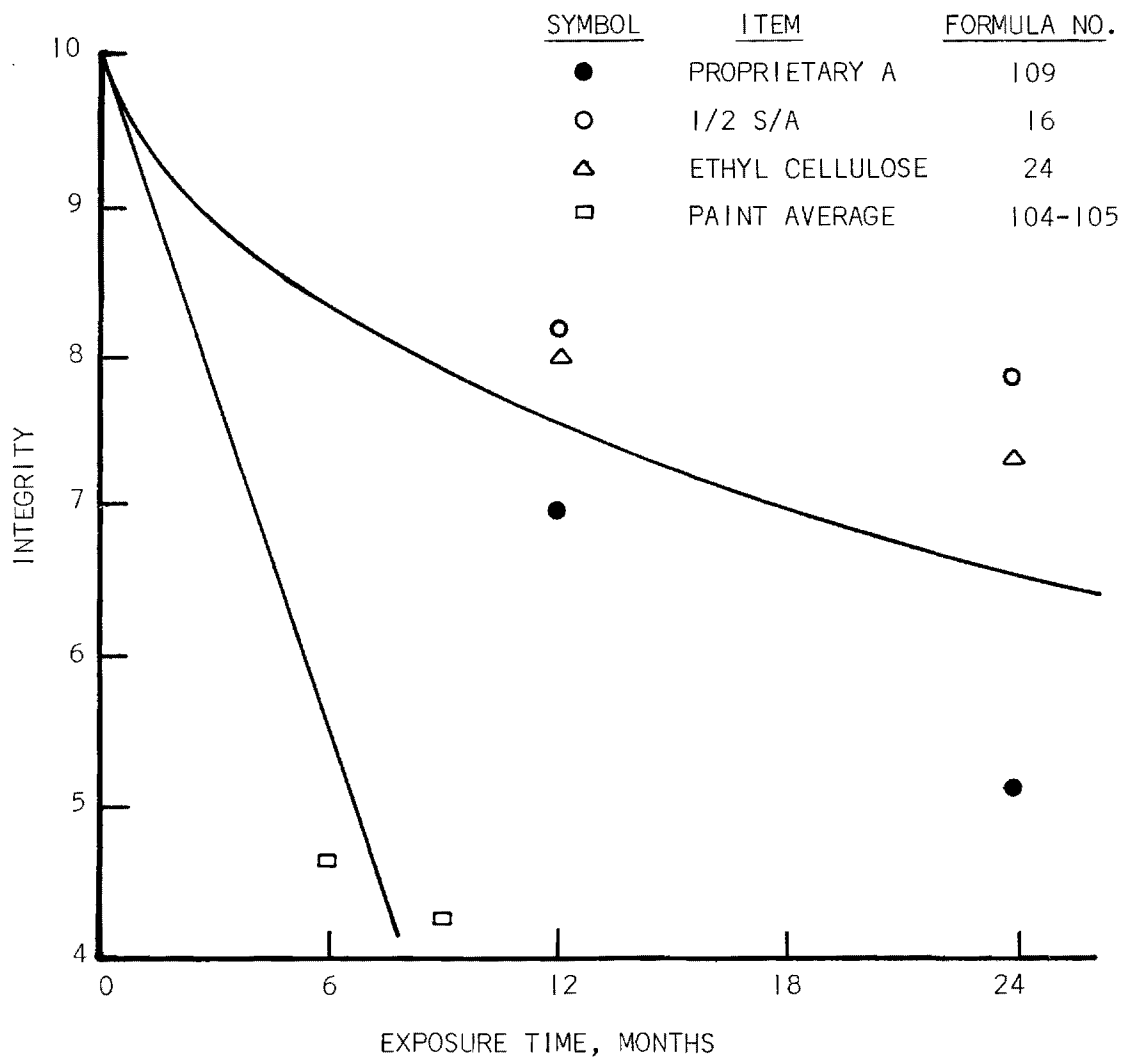


Figure 11. Integrity of hot melts and paint versus time, series I concrete only.

While integrity has been treated as the primary performance parameter, requirements of night visibility retention might impose more severe limitations on the useful life of the formulations here investigated. From an examination of Figure 12 one may observe that the hot melts undergo rapid initial loss of night visibility similarly to paints, but beyond one year they "plateau" at levels slightly below 10. This plateau may extend for the full integrity life of the film. Because of this plateau effect, we are unwilling to "write-off" the hot melts on the basis of an arbitrary minimum night visibility value. Certainly useful function is not totally lost, and the prospects of elevating the "plateau" are good.

We have noted, as shown in Figure 13, that a positive correlation exists between night visibility retention and application properties. It seems reasonable to expect that formulations which exhibit superior flow in application may also wet and secure drop-on beads more effectively. We have also found some evidence (in part from Series II) that the "toughness" as measured by impact tests is higher for the formulations of superior night visibility retention. Certainly this quality of the resin matrix must contribute to its bead-bonding capabilities.

The principal findings of Series I are summarized as follows:

1. The integrity performance of several types of epoxy-primed hot melts on P/C concrete is so superior to paint that meaningful comparisons are not possible within the period of wear testing.

2. The night visibility retention of hot melts is greatly superior to paints, but the level of the visibility retained is marginally useful, therefore priority attention to this limitation is indicated for any future R. & D. activity.

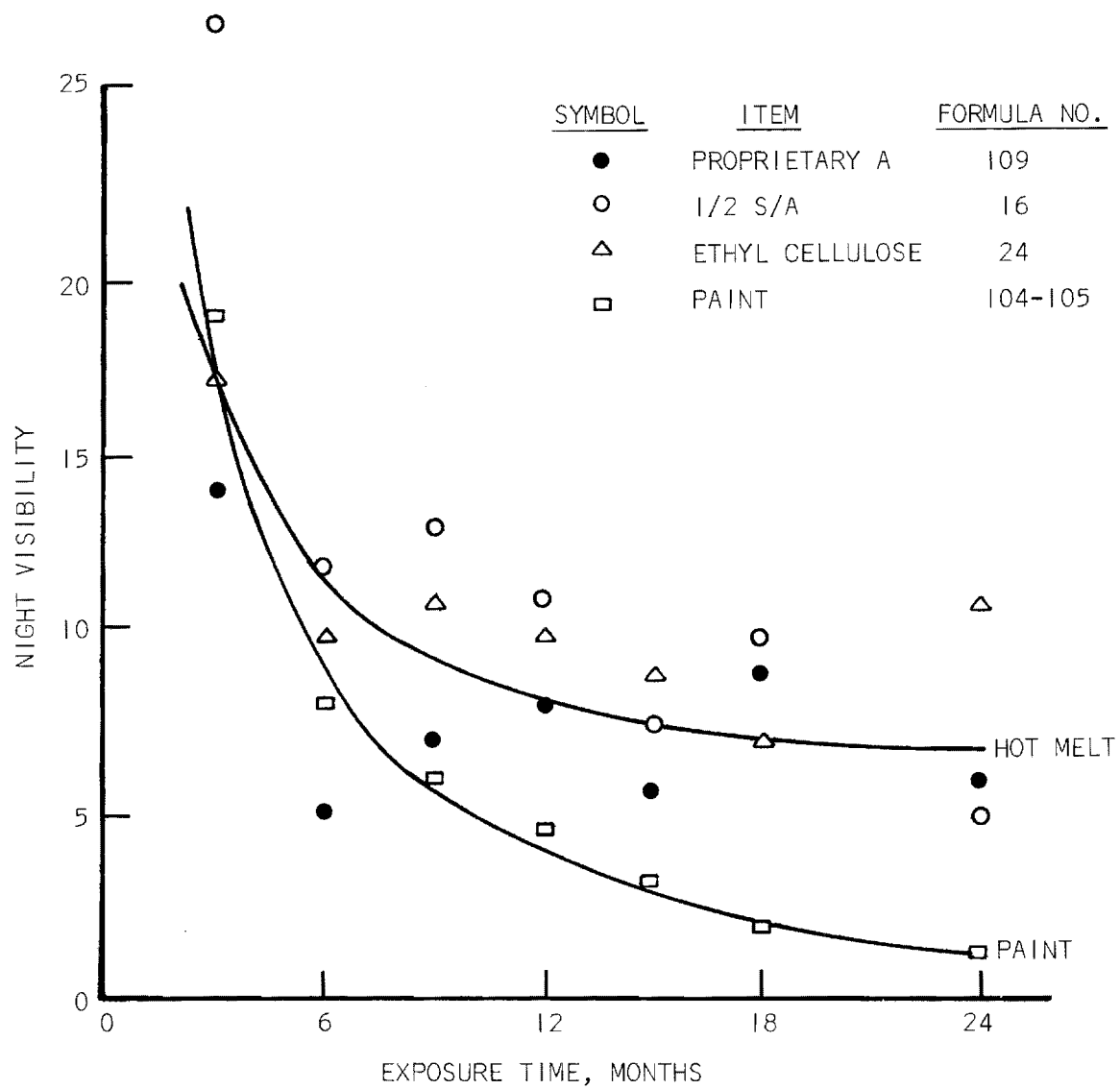


Figure 12. Night visibility of hot melt and paint versus time, series I concrete only.

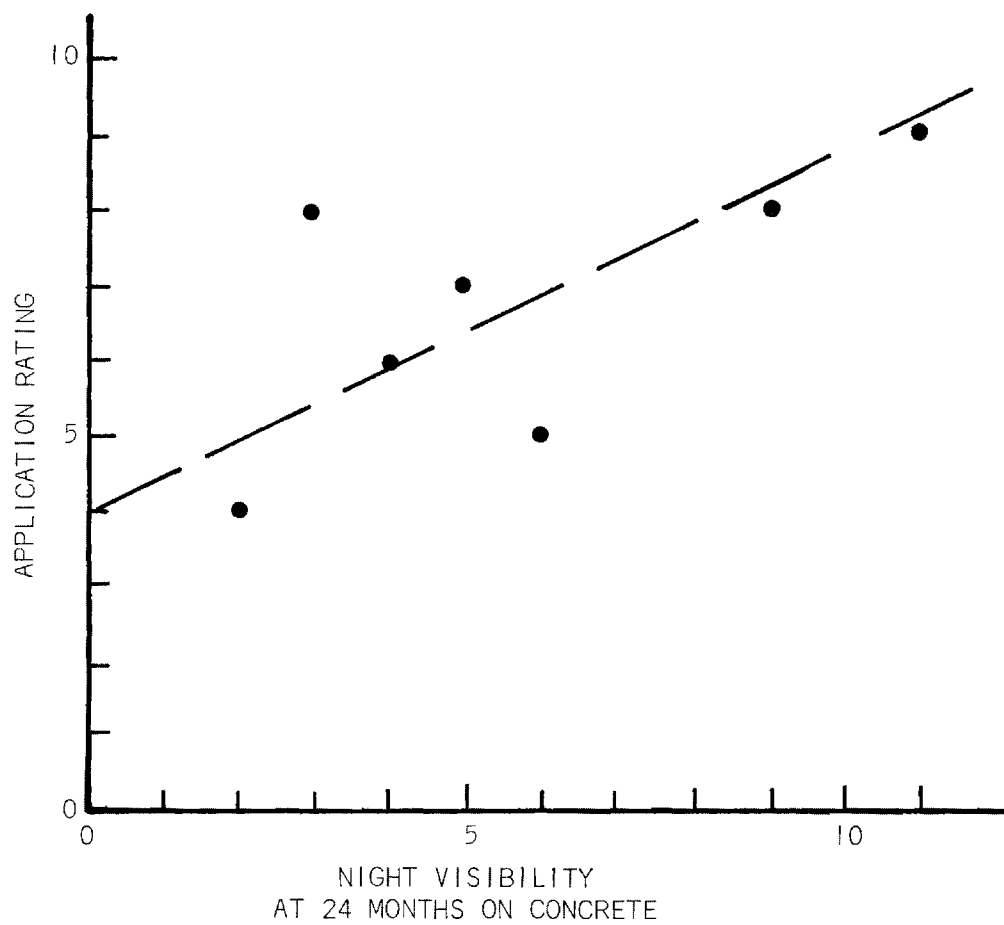


Figure 13. Correlation of night visibility with application properties.

3. Application thickness of 3/32" are not quite adequate for satisfactory application and performance with existing materials and methods. Very satisfactory application and performance is attained at 3/16" thickness. Optimum cost-effectiveness should be realized at about 1/8" thickness.

D. Highway Tests, Series II

1. Purpose

Much laboratory formulation and testing work was pursued in the one year interval between placements of Series I and II. Since the results of Series I were not available during this period, efforts were being directed primarily towards finding means of improving physical properties and/or reducing materials costs. Interest finally centered on certain polyamide materials for the former purpose and on non-drying alkyds for the latter. One purpose of Series II was to seek field confirmation of these "advances" developed in the laboratory.

Series I was lacking in any systematic treatment of physical property variations that could provide reliable indications of performance relationship. This feature was to be included in Series II.

An idea had been developed for improving wet night visibility by corrugating the surfaces of hot melt stripes. The technique and the durability of the effect were to be evaluated within the Series II studies.

In summary, the purposes of Series II were to seek confirmation of advances in formulations, to further explore composition-physical property-performance relationships, and to evaluate wet night visibility enhancement by surface texturing.

2. Experimental Plan

Seven formulations were selected for use in Series II. These included a proprietary, the California Spec., two alkyds, and three polyamides. The compositions are summarized in Table XIII. One control paint was used, the standard alkyd identical with the one used in Series I.

Series I provided much guidance for design decisions. Applications to both concrete and asphalt substrates were definitely indicated. Epoxy had been found to be the preferred primer for concrete, but priming of asphalt was not seen to be especially useful. Film thickness was fixed at a level of 3/16" gate clearance corresponding to the heavier applications of Series I. Surface corrugation of hot melts was investigated to determine if this method of enhancing wet night visibility would affect durability.

The experimental design as analyzed factorily is shown in Table XIV. The corrugation effect and paint performance were examined separately.

3. Placements and Observations

Applications of Highway Series II were made on Interstate 85 at Lenox Road Bridge on May 31, 1966. The site location was contiguous with the prior placements of Series I. In addition, three stripes were applied to the asphaltic substrate of the parking lot between the Architecture and E.E. Buildings on the Georgia Tech Campus. These stripes were Stripe #454 Beaded Hot Melt #160, plain, Stripe #455 Beaded Hot Melt #160, corrugated, and Stripe #456 Beaded Traffic Paint #159. Altogether 56 stripes were applied. These included 24 on concrete, numbered 401-424, and 28 on asphalt, numbered 425-453.

The technique of corrugating stripes for these tests was crude but effective. The corrugation impressions were produced with a short length of 1 inch diameter pipe pressed manually into the hot bead-covered plastic stripe. A long handle welded to the impressor-pipe in the form of a "T" allowed the operator to apply impressions from a standing position. Impressions were applied transverse to the stripe length at intervals of approximately 6 inches. Each impression was generated with a slight forward as well as downward force on the pipe so that a slight mounding of thermoplastic material occurred immediately forward of the cylindrical depression. The corrugating operation followed-up the applicator machine at an average distance of about 6 feet. The correct distance must be judged by the quality of the impression. When the impressor follows too close, the hot thermoplastic tends to stick to the pipe; and when it is too far behind the applicator, the cooled thermoplastic may be too firm to be deformed.

Approximately two months after application, all highway placements on asphalt were obliterated by unexpected repaving operations. Accordingly, only the concrete and parking lot placements were left for further observations. The observation procedure was identical with that described for Series I, with all data recorded on IBM card formats. The final observations were at 12 months.

A special observation procedure was used on the parking lot placements. On the evening of June 17, 1966 these placements were photographed with 16mm movie equipment under headlight illumination at ranges of 250, 150, and 50 feet. Subsequently, the stripes were showered with water and photographed wet. The sequence of photographs is shown in Figure 14. As observed in the photographs, the left line is plain Hot Melt #160 Beaded,

TABLE XIII
EXPERIMENTAL FORMULATIONS FOR HIGHWAY SERIES II

		Hot Melt Formulation No.					
	Constituent	160	162	163	157	155	158
No.	Description	(Per Cent by Weight)					
197	Hydrogenated Rosin	5.79	-	-	-	-	-
88	S/A Resin	8.99	-	-	-	-	-
81	Hydrogenated Ester Gum	9.79	8.92	10.32	7.38	12.29	18.43
180	Alkyd Non-drying	-	8.92	8.84	-	-	-
114	Maleic Resin	-	6.72	5.39	-	-	-
217	Polyamide	-	-	-	12.90	9.21	4.66
212	Plasticizer	-	-	-	4.30	3.08	1.48
172	Std. Glass Beads	20.78	-	-	-	-	-
171	Sm. Glass Beads	-	25.14	25.14	25.14	25.14	25.14
16	Titanium Dioxide	13.09	3.50	3.50	3.50	3.50	3.50
85	Calcium Carbonate, Fine	41.56	21.64	21.64	21.64	21.64	21.64
159	Calcium Carbonate, Coarse	-	25.14	25.14	25.14	25.14	25.14

TABLE XIV
DESIGN OF SERIES II
FACTORIAL EXPERIMENTS

		Factorial Experiment		
		E	F	G
Materials:				
Proprietary C	(164)	X	X	
Cal. Spec.	(160)	X	X	
Low Polyamide	(158)	X	X	
Middle Polyamide	(155)	X	X	
High Polyamide	(157)	X	X	
Alkyd I	(161)	X	X	
Alkyd II	(162)	X	X	
Alkyd Paint	(159)			X
Thickness:				
3/16 in.		X	X	
10 mils				X
15 mils				X
20 mils				X
Priming:				
None		X	X	
Epoxy		X		
Substrate:				
Concrete		X	X	X
Asphalt			X	X
Surface Texture:				
None		X	X	
Corrugated			X	

the centerline is corrugated Hot Melt #160 Beaded, and the right line is Alkyd Paint #159 Beaded. At all ranges the middle (corrugated) line is obviously superior in visibility in the dry state. In the wet state it is the only visible line. The lines are not deeply inundated, but are wetted with a thin film of water. This completely quenches retro-reflection of the plain lines, but only slightly quenches retroreflection of the corrugated line.

4. Data Analysis and Discussion

Although 12 months on the highway is a short period for hot melts, some formulation insight is provided from the results of this test series. An analysis of variance of the uncorrugated stripes on concrete is tabulated in Appendix A. The results are summarized together with corrugation and paint performance data in Table XV. The findings are interpreted as follows:

Priming

Epoxy primer significantly enhances hot melt integrity performance on concrete. A corresponding inexplicable loss of night visibility performance was observed. On balance, the integrity improvement justifies the priming treatment.

Formulations

Most of the hot melts are so superior to paint in integrity and night visibility that comparisons at one year are not feasible. Among the hot melts, the distinctive features are the relative inferiority of integrity and application properties of #155 and #157 and the scattering of night visibility performance with #157 best and #164 worst. The poor application properties of #155 and #157 are undoubtedly caused by the

TABLE XV
ANALYSIS OF SERIES II, 12 MONTHS

	<u>Integrity</u>		<u>Night Visibility</u>		<u>Application Rating</u>
	<u>\bar{X}</u>	<u>95% C.L. ($\pm ts$)</u>	<u>\bar{X}</u>	<u>95% C.L. ($\pm ts$)</u>	
		\bar{X}		\bar{X}	
<u>Based on Anal. of Var.:</u>					
Primer:					
None	6.6	2.4	11.0	0.9	
Epoxy	8.8	2.4	8.0	0.9	
Formulations:					
155 M. Polyamide	6.0	4.0	8.5	1.6	5
157 H. Polyamide	5.5	4.0	13.5	1.6	4
158 L. Polyamide	8.0	4.0	9.0	1.6	6
160 Cal. Spec.	8.5	4.0	8.5	1.6	8
162 Alkyd I	8.0	4.0	11.5	1.6	7
163 Alkyd II	9.0	4.0	10.5	1.6	7
164 Proprietary C	9.0	4.0	5.0	1.6	8
<u>Averages Only:</u>					
All Plain Hot Melt, Unprimed	6.6		9.5		
All Corrugated, Unprimed	5.9		11.3		
#159 Standard Alkyd Paint	1.6		6.0		

polyamide component. Even though the viscosities of these materials were low as determined instrumentally at moderate shear rates, the flow-outs under the very low shear rates obtained in field applications were inadequate. A tendency for polyamides to impart low shear structure (as in gel paints) is well known, so, in retrospect, this effect is not entirely surprising. It emphasizes a need for low-shear viscosity characterization. The somewhat inferior integrity of the polyamide modified formulations is believed to be associated in part with the poor application properties. One may also speculate about the possible effects of other distinctive characteristics of the polyamides such as their relatively low static indentation properties. This again, might relate logically to the gel structure tendencies of the polyamides. It would seem that the most attractive enhancements of static and impact stress properties which are attainable with polyamide modification cannot be made useful in practice because of the foregoing difficulties. In fairness to the polyamides, we must note that the poor integrity averages resulted from serious failures on unprimed concrete. Very good integrity was observed on primed concrete. Thus, ultimately, a superiority might be demonstratable on primed concrete.

Good field performance of alkyd modifications was confirmed. Although #162 exhibited "harder" initial properties than #163 (lower creep and indentation), field performances were similar. Moreover, the relatively poor stress properties of these materials as demonstrated by laboratory tests strongly suggests that these properties are either irrelevant or distinctly secondary in importance (at least for exposure periods of up to two years). We have not been able to isolate any "critical" properties in terms of laboratory tests. It is our belief that bond failure is the

critical problem, that it develops cumulatively in service, but that thermoplastic formulations which exhibit "true" viscosity are able to "heal" any slight separations and thus maintain a more continuous bond. If this is a correct model, it explains the inadequacies of the tests utilized and suggests directions for further research. The previously mentioned low static indentation of the polyamide formulas is consistent with these concepts. Practical tests of low shear long term creep and of "cold-bonding" are suggested as potentially useful.

Despite the attainment of interesting stress properties with polyamides and economy and good durability with alkyd modification, the "California Spec " #160 was found to exhibit the best over-all balance of performance properties. Since the same rosin-derived resins, carefully selected for thermal stability, were used throughout all of the formulations of Highway Series II, the properties of the modifying resins or polymers are primarily responsible for relative performance. Consider the following:

1. Polyamide,
2. Non-drying Alkyd, and
3. Styrene-acrylate resin.

When specimens of these resins are heated simultaneously on a hot plate to temperatures of about 650° F both polyamide and alkyd begin to thicken and darken. The styrene-acrylate resin melts, depolymerizes and partially evaporates--leaving less of a dark residue. This is a most desirable form of degradation, as long as depolymerization is not excessive at melt temperatures, since it neither thickens nor darkens excessively the resin matrix.

Corrugation-Night Visibility

All corrugated hot melts were applied without priming to emphasize any integrity advantage or disadvantage of the corrugating effect. The result, as shown in Table XV, was that integrity was slightly impaired. The loss was not judged to be serious and priming would probably reduce the effect. The instrumentally determined night visibility of corrugated hot melts is somewhat higher than plain hot melts. More significantly, however, the observed and photographed effects of wet night visibility retention of the corrugated stripe as presented in Figure 14 most emphatically illustrates the special merits of the corrugated treatment.

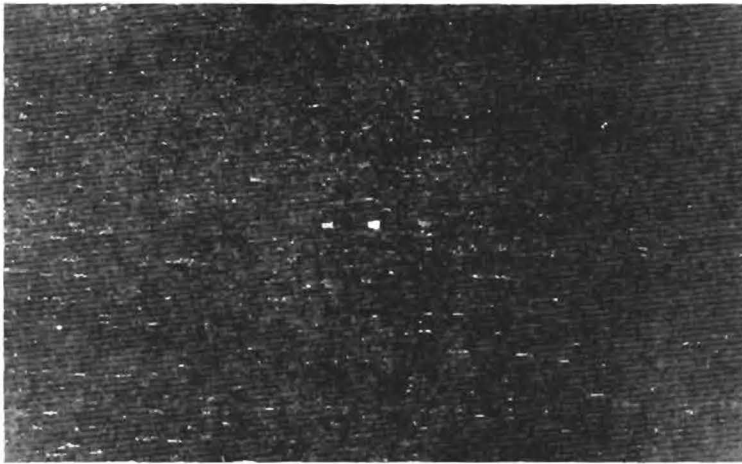
In summary, the findings of Highway Series II:

1. Discouraged the theory that high stress resistance formulations (polyamide modifications) would exhibit distinctly superior performance;
2. Confirmed the findings of good performance (at least equivalent to proprietary products) of hot melts based on blends of hydrogenated rosin esters with non-drying alkyds and especially with styrene-acrylate polymer; and
3. Demonstrated the practical value of corrugated surface texturing of hot melts as a means of attaining useful levels of wet night visibility of traffic stripes.

D. Economies of Materials and Applications

The validity of any economic assessment will depend upon the applicability of numerous assumptions, and final decisions must be conditioned by intangible factors to which costs or values cannot be objectively assigned.

This analysis is confined to comparisons of average annual costs per mile of striping based on the available materials, labor and equipment



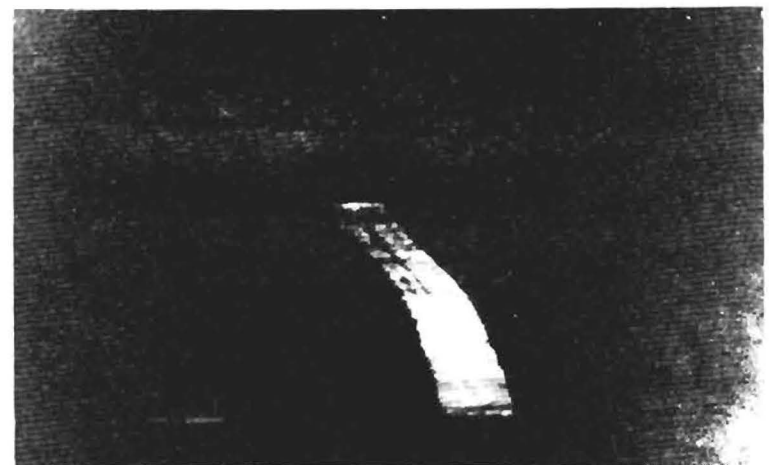
(a) RANGE 250 FT., DRY.



(b) RANGE 150 FT., DRY.



(c) RANGE 50 FT., DRY.



(d) RANGE 50 FT., WET.

Figure 14. Night visibility photographs.

amortization costs. The "life" of striping assumed is based on reasonable extrapolations from our research findings. In the case of the hot melts, a five year life is believed to be within the integrity capabilities of the material, although effective night visibility could not be expected to be maintained at the average levels that would be realized with five annual applications of beaded paint. No attempt has been made to evaluate safety or inconvenience factors to motorists. Interest costs, based on the time of application of maintenance funds, have not been included. In our judgment, the analysis is "realistic" and reflects neither undue optimism nor pessimism as to hot melt performance.

The findings are summarized in Table XVI, Economics of Hot Thermoplastic Traffic Stripes. Most of the formulations that have been road tested are included. In addition we have included a "Maximum Economy" formulation in which costs are substantially reduced by replacing glass beads in the formulation with coarse filler. While this modification has not been field-tested, it would certainly be entirely feasible. Finally, we show the comparison with traffic paint.

The results indicate that even the most economical hot thermoplastic composition is not quite fully competitive with traffic paint. The service life of hot thermoplastic should be at least $283/45 = 6.3$ times that of traffic paint for full economic parity. Some circumstances exist (such as urban cross-walks) where paint is simply unfeasible or inadequate. Thermoplastic is clearly indicated for such applications. At present, we cannot recommend general highway usage of thermoplastic markings. Such general usage might be justified when and if wet night visibility performance

TABLE XVI

ECONOMICS OF HOT THERMOPLASTIC TRAFFIC STRIPES

Basis:

	<u>Hot Thermoplastic</u>	<u>Paint</u>
Dimensions:	0.1 in. thickness x 4.0 in. wide x 1 mile long	15 mils wet thickness x 4.0 in. wide x 1 mile long
Beading:	6 lbs/300 ft length	6 lbs/300 ft (1 gal)
Priming:	Epoxy, 5 mils wet x 6 in. wide	None

<u>Formulation</u>	<u>All Materials*</u> <u>(\$/mile)</u>	<u>Placement*</u> <u>(\$/mile)</u>	<u>Total Installed</u> <u>(\$/mile)</u>	<u>Service Life</u> <u>(Yrs.)</u>	<u>Ave. Annual Cost</u> <u>(\$/mile)</u>
#26	397	9	406	5	81
#27	461	9	470	5	94
#28	380	9	389	5	78
#155	467	9	476	5	95
#157	538	9	547	5	109
#158	388	9	397	5	79
#160	397	9	406	5	81
#162	352	9	361	5	72
#163	352	9	361	5	72
Max. Economy	274	9	283	5	57
Proprietary A	1036	9	1040	5	208
Ga. Spec. Paint	43	2	45	1	45

* Detailed cost computations are given in Appendix C.

of textured lines becomes a proven capability.

Detailed computations in support of the data in Table XVI are presented in Appendix C.

VIII. SPECIFICATION DEVELOPMENT WORK

A. Laboratory-Field Correlations

The developmental testing data summarized in Tables XVII and XVIII were compared with field performance results in an effort to demonstrate possible empirical correlations. It was immediately evident that the range of field performance was too narrow to provide significant correlation information. This is, by no means, to be interpreted as evidence of irrelevance of the physical tests. It does suggest that acceptable ranges of some physical properties may be quite broad (but obviously not unlimited). Further, it imposes a need for greater application of rational judgement based on fundamental principles in selecting specification test requirements. Finally, this was seen to demand further attention to the development of additional specification tests having clearly defined and necessary relationships to service performance.

A review discussion of developmental test methods, their relationships to performance, and certain recognized limitations is given below as an introduction to the subsequent presentation of additional work on Acceptance Test Methods.

Consistency

This test presents several difficulties of technique. In attempting to measure consistencies while the material is allowed to cool, beads and coarse fillers tend to settle, so that the mixture may not remain homogenous. Also, a temperature gradient tends to develop in the material, and correct temperature measurement is rendered more difficult. These problems have been largely resolved by careful attention to technique. More troublesome, really, is the indication that viscosity as determined does not always characterize the flow of hot melt under application conditions. Apparently, viscosity must be measured at very low shear rates to correspond precisely with application conditions. Despite these limitations the consistency test is an essential characterization method.

TABLE XVII

PHYSICAL TESTS OF HIGHWAY TEST COMPOSITIONS, SERIES I

Hot Melt No.	Description	Density wt/gal	Consistency		Elastic Modulus (psiX10 ²)	Flexural Creep Properties			Static Shear Bond Strength			Flexural Impact Tests			
			@ 350° F (cps)	Slope @ 350° F (cps/°F)		Initial Elastic Deformation (in/inX10 ⁻³)	Creep Rate (in/in/minX10 ⁻³)	@ 77° F @ 0° F (psi) (psi)	Change (%)	Max. Fiber Stress @ 77° F (psi)	Absorbed Energy @ 77° F (in lb/in ²)	Max. Fiber Stress @ 0° F (psi)	Absorbed Energy @ 0° F (in lb/in ²)		
108	Proprietary B	17.3	7,500	390	3.8	1.5	5	-	-	-	1,630	1.65	2,100	2.7	
109	Proprietary A	18.1	22,400	330	2.3	2.0	12	395	45	-89	1,310	2.28	2,260	2.55	
26	Cal. Spec.	16.4	12,000	388	4.6	1.0	40	290	3	-99	2,310	2.94	2,870	4.05	
27	Ethyl Cellulose	16.5	1,600	48	9.2	0.5	2	45	1	-98	2,410	3.2	4,600	5.27	
28	Alkyd	16.9	7,300	40	4.6	1.0	10	135	8	-94	1,130	1.6	2,000	2.04	

TABLE XVIII

PHYSICAL TESTS OF HIGHWAY TEST COMPOSITIONS, * SERIES II

Hot Melt No.	Description	Density wt/gal	Consistency		Elastic Modulus (psiX10 ⁵)	Flexural Creep Properties			Static Shear Bond Strength			Flexural		Impact Tests	
			@ 350° F (cps)	Slope @ 350° F (cps/°F)		Initial Elastic Deformation (in/inX10 ⁻³)	Creep Rate (in/in/minX10 ⁻³)	@ 77° F (psi)	@ 0° F (psi)	Change (%)	Max. Fiber Stress @ 77° F (psi)	Absorbed Energy @ 77° F (in lb/in ²)	Max. Fiber Stress @ 0° F (psi)	Absorbed Energy @ 0° F (in lb/in ²)	
155	Middle Polyamide	16.39	27,000	260	0.35	13.0	150	0	500 ⁺	-	5,990	15.3	2,680	4.2	
157	High Polyamide	16.61	28,000	920	0.21	22.0	35	225	500	+120	-	>77	3,400	4.5	
158	Low Polyamide	16.3	25,000	180	1.8	2.5	35	400 ⁺	100	-75	1,770	2.6	2,040	2.5	
160	Cal. Spec.	16.7	34,000	720	4.6	1.0	135	380	340	-10	2,500	3.4	4,000	4.8	
162	Alkyd I	17.0	50,000	360	4.6	1.0	32	285	20	-93	1,230	2.0	2,220	3.2	
163	Alkyd II	17.0	44,000	480	2.3	2.0	390	250	150	-40	1,360	2.7	2,040	2.8	
164	Proprietary C	18.1	62,000	1,160	4.6	1.0	260	290	300	+03	2,320	3.5	2,270	3.0	

* These samples were drawn from the molten melts at the time they were being applied to the highway.

Flexural Creep Properties are of importance, although the requirements are not critical. Clearly, a very high creep rate would indicate excessive flow, with resultant spreading or smearing of lines, particularly in hot weather. Very low creep suggests possible "brittleness," but this would be inconclusive unless confirmed by other tests.

Elastic Deformation and Modulus have not been related directly to performance (except in an early theoretical study), but practical hot thermoplastics appear to be limited to a fairly small range of modulus values.

Static Shear Bond Strengths have not shown any relevance to field performance, nor have Flexural Impact Tests. It has been seen that these tests dramatically display the outstanding stress properties of polyamide modified formulations, but these properties have not been clearly demonstrated to be necessary for high performance in highway service. It is possible that an effect will be manifested on the highway after periods beyond the two year scope of this project. Polyamide modified formulations on epoxy primed concrete could display superior performance ultimately, and thus reflect their superior stress properties.

B. Acceptance Test Methods-Specification Development

1. Performance Versus Compositional Specifications

Accumulated experience indicates that hot melt compositions exhibiting satisfactory field durability can be prepared from numerous types of raw materials. The development of specifications requiring particular resin and plasticizer ingredients would appear to be unwise at the

present time for several reasons. A sufficient reason is that the "state of the art" has not yet been sufficiently developed that a specification of compositional limitations can be adequately supported by materials characterization methods and by field performance findings. Moreover, the use of a compositional specification would tend to discourage formulation research by suppliers and potential suppliers. Such research is needed. Most important, we feel that the requirements for hot melt traffic marking materials can be defined adequately by properly chosen laboratory performance tests. These requirements can be advanced, as desired, with advances in the state of the art so as to encourage maximum ingenuity in achieving improved performance and economy.

Performance specification test development was approached from a different point-of-view from much of the research and development testing pursued earlier. Research testing involved efforts to determine functional relationships of composition and physical properties. Specification testing is aimed at establishing minimum acceptable performance levels. In some cases equipment and procedures may be simplified, but at the same time, increased attention was directed to environmental factors of known significance.

2. Study of Hot Melt Heat Aging

A tendency of most hot melts to degrade in color and physical properties upon prolonged heating has been observed from the beginning of our formulation work. This factor has often been suspected as an important source of difficulties in reproducing physical test results. Accordingly, a decision was made to study the heat aging effect and to consider the

advisability of standardizing a heat aging treatment as a preliminary to physical testing so that the observed properties of hot melts would reflect their thermal stability.

Treatment conditions were chosen that were believed to be realistic with respect to current field practices.

Heat Aging Conditioning

Temperature	-	400 ± 10° F
Time	-	4 hours
Agitation	-	Slow, continuous, approx. 250 RPM

The material for treatment is melted in a heated 1 quart stainless steel beaker with a propeller agitator that is set close to the bottom. The beaker should be at least half filled with molten material. Timing begins when the specified temperature is reached. Test specimens should be prepared immediately following conditioning.

Physical test changes for several formulations before and after heat aging are given in Table XIX. The California Specification was found to be very stable in consistency, Proprietary C increases substantially, but all other hot melts are degraded to various degrees. Flexural creep tends to be reduced by heat aging except for the alkyds. Since the alkyds contain no volatile plasticizers, they cannot be stiffened by plasticizer losses, so they must be "self-plasticized" by alkyd degradation. Heat aging produces losses in impact properties in most cases. Proprietary C is again an outstanding exception--possibly this material undergoes some polymerization or condensation.

TABLE XIX
EFFECTS OF HEAT AGING

Hot Melt No.	Description	Consistency @ 350° F Change (%)	Flexural Creep Change (%)	Flexural Impact	
				Max. Fiber Stress @ 77° F Change (%)	Absorbed Energy @ 77° F Change (%)
155	Middle Polyamide	-4	0	-42	-81
157	High Polyamide	-19	-90	+26	-99
158	Low Polyamide	-24	-50	-11	-10
160	Cal. Spec.	0	-29	-34	-24
162	36% Alkyd	-39	+3000	+11	+6
163	42% Alkyd	-7	+36	-46	-20
164	Proprietary C	+98	-38	+31	+15

Regardless of the causes, significant changes of properties occur in all hot melts as a result of heat aging. Service application of hot melts may be expected to involve varying degrees of heat aging. Therefore, a standardized heat aging conditioning is an appropriate preliminary step for the characterization of hot melt compositions.

3. Test Procedures Development

It was proposed that for purposes of specification testing, all hot melts be subjected first to the Heat Aging Treatment. Test specimens would be prepared only from the heat-aged material. In this condition, the following tests would be performed:

1. Viscosity
2. Flexural Creep Rate
3. Static Indentation
4. Reflectance

Other specimens would be subjected to a Water-Freezing Cycle Treatment for the following tests:

1. Static Tensile Bond
2. Falling Ball Impact

It will be noted that these tests are performed on (a) frozen specimens at 0° F, and (b) frozen, thawed and water soaked specimens at 77° F. All test procedures are detailed in Appendix B as a part of a "Tentative Thermoplastic Traffic Stripe Specification."

A portion of the experimental work in support of this testing scheme is summarized in Table XX. By comparisons of "As Received" versus "Heat Aged" specimens, one notes that heat aging produces variable but often very

TABLE XX
SPECIFICATION DEVELOPMENT TESTS

Hot Melt No.	Description	"As Received"			Heat Aged			Water-Freezing Cycle				
		Consistency @ 350°F (cps)	Flexural Creep Rate (in/in/min $\times 10^{-3}$)	Static Indentation 200 gm (mils)	Consistency @ 350°F (cps)	Flexural Creep Rate (in/in/min $\times 10^{-3}$)	Static Indentation 200 gm (mils)	Reflectance (Green Filter)	Static Tensile		Falling Ball Impact	
									Bond @ 77°F (psi)	Bond @ 0°F (psi)	(Per cent unbroken after 3 impacts)	(Per cent unbroken after 3 impacts)
155	Middle Polyamide	4,600	3.0	47	4,400	3.0	78	53	200+	200+	100	100
157	High Polyamide	3,700	250	120	3,000	24	62	56	200+	200+	100	100
158	Low Polyamide	3,400	4.0	47	2,600	2.0	78	52	17	78	100	20
160	Calif. Spec.	26,500	7.0	140	26,500	5.0	110	76	15	61	100	100
162	Alkyd	11,400	4.0	190	7,800	130	510	37	26	45	60	20
163	Alkyd	7,500	35	230	7,000	55	470	49	2	56	100	0
164	Proprietary A	9,000	48	260	17,800	30	310	73	25	96	100	40
169	Calif. Spec. High Density	25,000	2.0	62	21,500	2.0	78	73	43	96	-	-
170	Alkyd Series I	24,000	13	94	18,000	300	340	73	40	85	60	0
171	Ethyl Cellulose	52,000	5	47	22,000	5	62	80	4	24	100	100
Tentative Specification Limits:												
	Maximum				35,000	30	200	-	-	-	-	-
	Minimum				2,000	-	-	74	-	-	80	80

significant effects on viscosity, creep, and static indentation; however, only the properties in the heat-aged condition are judged to be relevant to specifications. Judgments regarding acceptable levels of the test results are indicated at the bottom of Table XX. The tabulation clearly shows some of the destructive effects of the water freeze cycle on tensile bond and impact. Fairly good bonds are the rule in the frozen condition, but subsequent thawing seriously disrupts bond integrity. On the other hand, impact resistance is reduced by freezing, but recovers upon thawing.

The above described test procedures and findings were believed to provide an adequate basis for product specifications. It is possible that formulations of superior performance may be developed eventually which would not conform in all respects to the presently proposed requirements. Once performance proof is forthcoming, then specification modifications would certainly be appropriate. In the meantime, we can affirm at least that formulations of known good performance will meet the proposed specifications, and it is improbable that poor products will conform with the requirements. A draft of proposed specifications is included in Appendix B.

IX. CONCLUSIONS AND RECOMMENDATIONS

The principal conclusions of this investigation are summarized as follows:

1. The integrity service life of properly applied hot thermoplastic traffic stripes is at least 3 and possibly 10 times that of good traffic paints.
2. The night visibility service life of hot thermoplastic traffic stripes is superior to traffic paints, but the margin of superiority is lower than for integrity.
3. Significant levels of wet night visibility are attainable by a surface texturing or corrugation process applied to hot thermoplastic stripes. Without such texturing, neither hot thermoplastics nor paint exhibit any appreciable wet night visibility.
4. Priming is essential for adequate performance of hot thermoplastics on P/C concrete paving; it was not demonstrated to be required on asphalt pavings. Clear epoxy resin formulations are preferred primers.
5. Surface preparation--clean dry pavement--is necessary for satisfactory hot thermoplastic stripe performance.
6. Film thickness of approximately 1/8 inch appears to be optimum for hot thermoplastic stripes.
7. An analysis of comparative economics indicates that hot thermoplastic stripes must exhibit at least 6.3 times the service life of traffic

paint to be competitive in average annual cost. This ratio represents an optimistic projection of thermoplastic costs. Recent contract terms for thermoplastic installation would require a thermoplastic life of 39 times that of traffic paint.

The following general recommendations are submitted:

1. The technique of surface texturing or corrugation of thermoplastics should be investigated on a practical service performance scale to ascertain durability and to evaluate subjectively the wet night visibility enhancement.

2. Further laboratory investigations need to be directed primarily to the development and interpretation of test methods for both research and quality control. Research on bead retention by thermoplastics is another important need.

3. Selective service use of hot thermoplastic striping is recommended. Areas of especially heavy traffic are suggested as offering the best conditions for acceptable relative economy.

APPENDIX

- A. Research Supporting Data
- B. Test Methods and Specifications
- C. Cost Calculations
- D. References Cited

A. Research Supporting Data

Table XXI Compositions of Experimental Formulations

Table XXII Paint and Primer Formulations

Table XXIII Experimental Formulations Physical Data

Table XXIV Constituents Lists

Analysis of Variance Data

TABLE XXI
COMPOSITIONS OF EXPERIMENTAL FORMULATIONS

Constituents		Hot Melt Formulation No.																
No.	Description	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17
		(Per Cent by Weight)																
81	Hydrogenated Ester Gum	9.8					16.0		20.5	9.8			14.3	5.8	9.8	9.8	14.3	12.3
197	Hydrogenated Rosin	5.8	33.3	9.3	9.3	5.8		5.8		5.8	5.8	5.8	5.8	14.3	5.8	5.8	5.8	
88	3/A Resin	9.0	36.7	14.5	14.5	9.0		9.0		9.0	9.0	9.0	4.5	18.0		9.0	4.5	
268	Petroleum Resin		40.0															
69	Petroleum Resin			15.7							9.8							
267	Petroleum Resin				15.7			9.8										
117	Petroleum Resin					9.8						9.8						
109	Ethyl Cellulose						5.0								9.0			
269	Ethyl Cellulose								6.7									
300	Plasticizer Resin								2.7									
92	Alkyd Non-drying																	12.3
270	Castor Oil #3						3.6											
39	Tricresylphosphate						0.2											
116	Blown Castor Oil #5								3.0									
271	Stabilizer A-5								0.2									
94	Pentylphenol								0.2									
107	Regular Glass Beads	20.8		16.7	16.7	20.8	20.8	20.8		20.8	20.8	20.8	20.8	20.8	20.8	20.8	20.8	20.8
16	Titanium Dioxide	13.0		11.4	10.4	13.0	13.0	13.0	3.3	13.0	13.0	13.0	13.0	13.0	13.0	13.0	13.0	13.0
85	Calcium Carbonate, Fine	41.6		33.4	33.4	41.6	41.4	41.6		41.6	41.6	41.6	41.6	41.6	41.6	41.6	41.6	41.6
95	Barytes								63.4									
Total		100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0

(Continued)

TABLE XXI (Continued)
COMPOSITIONS OF EXPERIMENTAL FORMULATIONS

Constituents		Hot Melt Formulation No.																	
No.	Description	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	
		(Per Cent by Weight)																	
81	Hydrogenated Ester Gum		0.8	9.9		9.8	9.8	9.8	9.8	9.8	9.8	9.8	12.3	9.8	11.5	4.9	4.5	6.6	
197	Hydrogenated Rosin		5.8					5.6	5.8	5.8	5.6		7.3	5.8	6.9				
88	S/A Resin		18.0		9.0				9.0	9.0			11.3	9.0	11.8				
92	Alkyd Non-drying	12.3		9.9															
114	Maleic Resin	12.3		4.9		4.9						4.9							
117	Petroleum Resin				15.6														
109	Ethyl Cellulose						9.1	9.1			9.1								
272	Pentylphenol						0.1	0.1			0.1								
180	Alkyd Non-drying					9.8						9.8				9.8	8.8	13.7	
175	Maleic Resin															9.8	8.8	13.7	
176	Polypropylene Gum																2.4	3.8	
116	Castor Oil						5.5												
107	Regular Glass Beads	20.8	20.8	20.8	20.8	20.8	20.8	20.8	31.2	20.8	20.8	20.8							
171	Small Glass Beads								15.6				24.2	21.8	23.4	25.2	25.2	38.8	
16	Titanium Dioxide	13.0	13.0	12.8	13.1	13.0	13.0	13.0	13.0	13.0	13.0	13.0		13.1	3.6	3.5	3.5	5.4	
159	Calcium Carbonate, Coarse												24.2	21.8	23.4	25.2	25.2		
85	Calcium Carbonate, Fine	41.6	41.6	41.7	41.5	41.7	41.7	41.6	15.6	41.6	41.6	41.7	20.7	18.7	19.4	21.6	21.6		
169	Asbestos Shorts																	18.0	
Total		100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	

(Continued)

TABLE XXI (Continued)
COMPOSITIONS OF EXPERIMENTAL FORMULATIONS

Constituents		Hot Melt Formulation No.																
No.	Description	35	36	37	38	39	40	41	42	43	44	45	46	47	48	49	50	51
		(Per Cent by Weight)																
81	Hydrogenated Ester Gum	40.0	8.9	11.1	8.9	9.8	8.9	8.9	9.8	8.9	8.9	8.9	8.9	8.9	15.1	8.9	8.9	8.8
197	Hydrogenated Rosin													2.2				
88	S/A Resin												2.2					
114	Maleic Resin	20.0	6.6	4.4	4.4	4.8	4.4	4.4	4.8	4.4	4.4	4.4	4.4	4.4	7.6	4.4	4.4	4.4
180	Alkyd Non-drying	40.0	8.9	8.9	11.1	9.8	8.9	8.9	9.8	8.9	8.9	8.9	8.9	8.9	15.1	8.9	8.9	8.9
178	Alkyd Non-drying							2.2										
176	Polypropylene Gum									2.2					4.1			
273	Urethane Elastomer						2.2											
109	Ethyl Cellulose										2.2							
274	Epoxy Resin											2.2						
275	Polyamide															2.2		
182	Wetting Agent														5.7			
191	SAIB																2.2	
276	Sulfur																	2.2
107	Regular Glass Beads																	
171	Small Glass Beads		25.2	25.2	25.2	25.2	25.2	25.2	25.2	25.2	25.2	25.2	25.2	25.2	43.0	25.2	25.2	25.2
169	Asbestos Shorts														8.5			
16	Titanium Dioxide		3.5	3.5	3.5	3.5	3.5	3.5	3.5	3.5	3.5	3.5	3.5	3.5	6.0	3.5	3.5	3.5
159	Calcium Carbonate, Coarse		25.2	25.2	25.2	25.2	25.2	25.2	25.2	25.2	25.2	25.2	25.2	25.2		25.2	25.2	25.2
85	Calcium Carbonate, Fine		21.7	21.7	21.7	21.7	21.7	21.7	21.7	21.7	21.7	21.7	21.7	21.7		21.7	21.7	21.7
Total		100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0

(Continued)

TABLE XXI (Continued)
COMPOSITIONS OF EXPERIMENTAL FORMULATIONS

Constituents		Hot Melt Formulation No.																
No.	Description	52	53	54	55	56	57	58	59	60	61	62	63	64	65	66	67	68
(Per Cent by Weight)																		
81	Hydrogenated Ester Gum	8.9	8.0	12.1	9.6	10.9	24.4	9.8	40.0	9.8	39.8	9.8	9.4	40.0	36.3	36.3	36.3	36.3
197	Hydrogenated Rosin		4.6		5.6	6.2			23.0	5.7	23.6	5.6	5.5					
88	S/A Resin										36.6	9.0	8.6		9.1			
114	Maleic Resin	4.4												20.0	18.3	18.3	18.3	18.3
180	Alkyd Non-drying	8.9		9.8				9.8						40.0	36.3	36.3	36.3	36.3
178	Alkyd Non-drying																	
277	Polycarbonate Resin	2.2		2.5												9.1		
109	Ethyl Cellulose		5.5		6.7	7.4			35.8	8.8								
176	Polypropylene Gum		2.0		2.4													
278	Butylphenol		0.1		0.1				1.2	0.3								
216	Zinc Resinate							4.8										
192	Epoxy Resin																9.1	
279	Polyester																	9.1
171	Small Glass Beads	25.2	26.6	25.2	25.2	25.2	25.2	25.2				25.2	25.6					
16	Titanium Dioxide	3.5	3.6	3.5	3.5	3.5	3.5	3.5		13.0		3.5	3.5					
159	Calcium Carbonate, Coarse	25.2	26.6	25.2	25.2	25.2	25.2	25.2				25.2	25.6					
85	Calcium Carbonate, Fine	21.7	23.0	21.7	21.7	21.7	21.7	21.7		41.6		21.7	21.8					
107	Regular Glass Beads									20.8								
Total		100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0

(Continued)

TABLE XXI (Continued)
COMPOSITIONS OF EXPERIMENTAL FORMULATIONS

Constituents		Hot Melt Formulation No.																
No.	Description	69	70	71	72	73	74	75	76	77	78	79	80	81	82	83	84	85
		(Per Cent by Weight)																
81	Hydrogenated Ester Gum	15.8	18.8	15.8	15.8	15.8	15.8	12.1	14.0		15.3	13.2	9.9	9.9	13.0	14.0	14.5	12.3
197	Hydrogenated Rosin				4.9	4.9	4.9	5.6	4.0		4.7	4.0	5.7		2.0	2.4	2.3	
88	S/A Resin	3.7	4.3		3.7							3.7					2.1	
114	Maleic Resin																	
180	Alkyd Non-drying	4.9		4.9											4.9	4.9	4.9	
191	Epoxy Resin		5.8			3.7			6.6			3.7			3.3	1.8	2.9	
279	Polyester			3.7			3.7											
277	Polycarbonate Resin														1.2	1.3	1.3	
194	Cellulose Acetate Butyrate							6.6										
190	Tall Oil Fatty Acids									27.4								
281	Phosphoric Acid									0.3								
195	Lime									2.2								
283	Calcium Acetate									0.1								
206	Polyester																	2.4
284	Epoxy Resin										3.5							
215	Wax																	9.8
193	Cellulose Acetate Butyrate												9.1	9.1				
191	SAIB														5.7			
196	Stabilizer												0.1	0.1				
278	Butylphenol																	
171	Small Glass Beads	25.2	23.7	25.2	25.2	25.2	25.2	25.2	25.2	30.0	25.4	25.2	25.1	25.1	25.2	25.2	24.2	25.2
16	Titanium Dioxide	3.5	3.3	3.5	3.5	3.5	3.5	3.8	3.0	4.2	3.6	3.5	3.5	3.5	3.5	3.5	3.5	3.5
159	Calcium Carbonate, Coarse	25.2	23.7	25.2	25.2	25.2	25.2	25.2	25.2	30.0	25.4	25.2	25.1	25.2	25.2	25.2	24.2	25.2
85	Calcium Carbonate, Fine	21.7	20.4	21.7	21.7	21.7	21.7	21.5	22.0	5.8	22.1	21.5	21.5	21.5	21.7	21.7	21.0	21.6
Total		100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0

(Continued)

TABLE XXI (Continued)
COMPOSITIONS OF EXPERIMENTAL FORMULATIONS

Constituents		Hot Melt Formulation No.																	
No.	Description	86	87	88	89	90	91	92	93	94	95	96	97	98	99	100	101	102	
		(Per Cent by Weight)																	
81	Hydrogenated Ester Gum	17.4		7.9	10.6	15.6	9.7	63.4	64.6	64.6									
197	Hydrogenated Rosin		63.5	7.9	5.3		2.9		20.2	20.2									
88	S/A Resin																		
114	Maleic Resin																		
180	Alkyd Non-drying																		
206	Polyester					9.0													
207	Polyester	10.1	36.5	9.2	9.1			36.1			25.0						20.0		
209	Polyamide											25.0	22.5	22.5	20.0	20.0			
284	Epoxy Resin						5.4												
192	Epoxy Resin								14.7	14.7									
285	Medium Length Chopped Fiberglass								0.5	0.5									
286	Finely Chopped Fiberglass						0.4	0.5											
287	Fine Sand						54.9												
171	Small Glass Beads	24.0		25.0	25.0	25.1	19.7				25.0	25.0	25.0	25.0	25.0	25.0	25.0		
16	Titanium Dioxide	3.4		3.8	3.7	3.5	7.0				3.5	3.5	3.5	3.5	3.5	3.5	3.5		
159	Calcium Carbonate, Coarse	24.0		25.0	25.0	25.1					25.0	25.0	25.0	25.0	25.0	25.0	25.0		
85	Calcium Carbonate, Fine	21.1		21.2	21.3	21.7					21.5	21.5	21.5	21.5	21.5	21.5	21.5		
212	Plasticizer												2.5		5.0		5.0		
151	Chlorinated Polyphenyls													2.5		5.0			
Total		100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	

(Continued)

TABLE XXI (Continued)
COMPOSITIONS OF EXPERIMENTAL FORMULATIONS

Constituents		Hot Melt Formulation No.														
No.	Description	105	106	107	108	109	110	111	112	113	114	115	116	117	118	119
(Per Cent by Weight)																
206	Polyester						100.0	80.0	90.0		90.0			91.0		80.0
207	Polyester									91.0		91.0	80.0		80.0	
212	Plasticizer											9.0	20.0	9.0	20.0	20.0
204	Chlorinated Polyphenyl	Paint	Primer	Primer			20.0	10.0		10.0						
151	Chlorinated Polyphenyl							9.0								
90	Proprietary Hot Melt A					100.0										
89	Proprietary Hot Melt B				100.0											
Total					100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0

(Continued)

TABLE XXI (Continued)
COMPOSITIONS OF EXPERIMENTAL FORMULATIONS

Constituents		Hot Melt Formulation No.																
No.	Description	120	121	122	123	124	125	126	127	128	129	130-1	130-2	130-3	131	132-1	132-2	132-3
		(Per Cent by Weight)																
81	Hydrogenated Ester Gum			12.2		24.7	12.4	16.5	18.6			50.0	33.0	20.0				
197	Hydrogenated Rosin																	
208	Polyamide		17.5	8.8											19.7			
209	Polyamide				29.7		14.8	9.8	7.4	17.1						77.0	77.0	77.0
212	Plasticizer	30.0	7.5	3.7	3.3		1.6	1.1	0.8		7.4				4.8			
206	Polyester	70.0									17.3	50.0	67.0	80.0				
204	Chlorinated Polyphenyls																	
151	Chlorinated Polyphenyls								7.4									
294	Plasticizer															23.0		
295	Plasticizer																23.0	
296	Plasticizer																	23.0
171	Small Glass Beads	25.0	25.1	22.3	25.1	23.7	24.2	24.4	25.2	25.1					25.2			
16	Titanium Dioxide	4.6	4.1	3.2	3.5	3.4	3.4	3.4	3.5	3.5					3.5			
159	Calcium Carbonate, Coarse	25.0	25.1	22.3	25.1	23.7	24.2	24.4	25.2	25.1					25.2			
85	Calcium Carbonate, Fine	20.4	21.0	19.2	21.6	20.4	20.8	21.0	21.6	21.6					21.6			
Total		100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0

(Continued)

TABLE XXI (Continued)
COMPOSITIONS OF EXPERIMENTAL FORMULATIONS

Constituents		Hot Melt Formulation No.																
No.	Description	132-4	132-5	132-6	132-7	132-8	132-9	132-10	132-11	132-12	132-13	133-1	133-2	133-3	133-4	134	135	136
		(Per Cent by Weight)																
81	Hydrogenated Ester Gum				23.0												2.5	6.2
206	Polyester											77.0	77.0	77.0	77.0	17.4	15.7	12.6
209	Polyamide	77.0		77.0	77.0	77.0	77.0	77.0	77.0	77.0	77.0							
212	Plasticizer															7.5	6.8	6.2
297	Plasticizer	23.0																
215	Wax			23.0														
270	Castor Oil #3					23.0						23.0						
216	Zinc Resinate						23.0						23.0					
280	Plasticizer							23.0										
282	DOP								23.0						23.0			
298	Petroleum Plasticizer									23.0								
299	Paraffin Wax										23.0							
294	Plasticizer													23.0				
171	Small Glass Beads															24.8	24.8	25.0
16	Titanium Dioxide															4.7	4.6	5.0
159	Calcium Carbonate, Coarse															24.8	24.8	25.0
85	Calcium Carbonate, Fine															20.8	20.8	20.0
Total		100.0		100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0

(Continued)

TABLE XXI (Continued)
COMPOSITIONS OF EXPERIMENTAL FORMULATIONS

Constituents		Hot Melt Formulation No.																
No.	Description	137	138	139	140	141	142	143	144	145	146	147	148	149	150	151	152	153
		(Per Cent by Weight)																
206	Polyester	12.6	12.6															
208	Polyamide			18.5	18.5	9.3	9.3	9.3										
81	Hydrogenated Ester Gum						15.4	15.4						9.1	11.3	9.1	10.0	5.6
181	Alkyd Non-drying													11.3	9.1	9.1	10.0	12.0
114	Maleic Resin		6.2											4.5	4.5	6.8	4.9	7.3
212	Plasticizer	6.2	6.2	6.2	6.2	15.4												
215	Wax	6.2																
171	Small Glass Beads	25.0	25.0	25.1	25.1	25.1	25.1	25.1						25.0	25.0	25.0	25.0	25.0
159	Calcium Carbonate, Coarse	25.0	25.0	25.1	25.1	25.1	25.1	25.1						25.0	25.0	25.0	25.0	25.0
16	Titanium Dioxide	5.0	5.0	3.5	3.5	3.5	3.5	3.5						3.5	3.5	3.5	3.5	3.5
85	Calcium Carbonate, Fine	20.0	20.0	21.6	21.6	21.6	21.6	21.6						21.6	21.6	21.6	21.6	21.6
Total		100.0	100.0	100.0	100.0	100.0	100.0	100.0						100.0	100.0	100.0	100.0	100.0

(Continued)

TABLE XXI (Continued)
COMPOSITIONS OF EXPERIMENTAL FORMULATIONS

Constituents		Hot Melt Formulation No.																
No.	Description	154	155	156	157	158	159	160	161	162	163	164	165	166	167	168	169	170
		(Per Cent by Weight)																
81	Hydrogenated Ester Gum	8.2	12.3	15.4	7.4	18.4		9.8		8.9	8.9		9.8		9.8	9.8	9.8	9.8
180	Alkyd Non-drying	11.0								8.9	10.3							9.8
114	Maleic Resin	5.8								6.8	5.4							4.9
217	Polyamide		9.2	9.2	12.9	4.7												
212	Plasticizer		3.1		4.3	1.5												
197	Hydrogenated Rosin							5.8					5.8		5.8	5.8	5.8	
88	S/A Resin							9.0					9.0		9.0	9.0	9.0	
223	Proprietary Hot Melt A											100.0		100.0				
171	Glass Beads	25.0	25.1	25.1	25.1	25.1				25.1	25.1						23.7	
107	Regular Glass Beads							20.8					20.8		20.8	20.8		20.8
16	Titanium Dioxide	3.5	3.5	3.5	3.5	3.5		13.1		3.5	3.5		13.1		13.1	13.1	7.5	13.0
159	Calcium Carbonate, Coarse	25.0	25.1	25.1	25.1	25.1				25.1	25.1						23.7	
85	Calcium Carbonate, Fine	21.5	21.7	21.7	21.7	21.7		41.5		21.7	21.7		41.5		41.5	41.5	20.5	41.7
Total		100.0	100.0	100.0	100.0	100.0		100.0		100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0

(Continued)

TABLE XXI (Concluded)
COMPOSITIONS OF EXPERIMENTAL FORMULATIONS

Constituents		Hot Melt Formulation No.		
No.	Description	171	172	173
		(Per Cent by Weight)		
81	Hydrogenated Ester Gum	9.8	12.4	9.3
109	Ethyl Cellulose	9.1		
180	Alkyd Non-drying		12.4	
197	Hydrogenated Rosin	5.6		
272	Stabilizer	0.1		
114	Maleic Resin		6.2	
217	Polyamide			16.3
212	Plasticizer			5.4
171	Glass Beads	20.8		
16	Titanium Dioxide	13.0	16.4	16.4
85	Calcium Carbonate	41.6	52.6	52.6
Total		100.0	100.0	100.0

TABLE XXII
PAINT AND PRIMER FORMULATIONS

Constituents		Hot Melt Formulation No.				
No.	Description	104	105	106	107	159
(Per Cent by Weight)						
73	Alkyd Drying		20.6			
289	Alkyd Drying	37.2				37.2
152	Chlorinated Rubber 10 cps		3.3			
63	Calcium Carbonate, Fine	14.9	33.0			14.9
15	Magnesium Silicate	9.9	6.0			9.9
130	Clay Thickener	0.4	0.3			0.4
17	Diatomaceous Silica	9.9	6.0			9.9
290	Aliphatic Solvent	11.3				11.3
122	Toluol		14.2			
32	Driers	1.5	1.5			1.5
150	Epichlorhydrin		0.2			
16	Titanium Dioxide	14.9	14.9			14.9
112	Rubber Adhesive			100.0		
291	Epoxy Resin				33.3	
292	Polyamide				16.6	
293	Acetone				50.1	
Total		100.0	100.0	100.0	100.0	100.0

TABLE XXIII
EXPERIMENTAL FORMULATIONS PHYSICAL DATA

Formulation Number	Density (Wt/Gal)	Consistency @ 350° F (cps)	Creep Rate (in/in/min x 10 ⁻³)	Static Shear Bond		Flexural Impact @ 77° F		Flexural Impact @ 0° F		Vehicle Consistency @ 350° F (cps)
				@ 77° F (psi)	@ 0° F (psi)	Max. Fiber Stress (psi)	Absorbed Energy (in-lb/in ²)	Max. Fiber Stress (psi)	Absorbed Energy (in-lb/in ²)	
1		28,000								1,700
2										2,300
3		62,000								1,800
4		48,000								2,000
5		17,500								1,450
6		13,200								650
7		36,000								2,000
8		7,500								3,000
9*										
10*										
11*										
12*										
13*										
14*										
15	16.4	12,000	40	290	3	2310	2.94			
16*										
17*										
18*										
19*										
20*										
21		13,000								
22	16.9	7,300	10	139	8	1130	1.6	2000	2.04	
23										
24	16.5	1,600	2	45	1	2410	3.2	4600	5.27	
25		5,500								
26	16.4	12,000	40	290	3	2310	2.94	2870	4.05	
27	16.5	1,600	2	45	1	2410	3.2	4600	5.27	

* These exploratory formulations were evaluated subjectively and not characterized by quantitative tests.

(Continued)

TABLE XXIII (Continued)
EXPERIMENTAL FORMULATIONS PHYSICAL DATA

Formulation Number	Density (Wt/Gal)	Consistency @ 350°F (cps)	Creep Rate (in/in/min $\times 10^{-3}$)	Static Shear Bond		Flexural Impact @ 77°F		Flexural Impact @ 0°F		Vehicle Consistency @ 350°F (cps)
				@ 77°F (psi)	@ 0°F (psi)	Max. Fiber Stress (psi)	Absorbed Energy (in-lb/in ²)	Max. Fiber Stress (psi)	Absorbed Energy (in-lb/in ²)	
28	16.9	7,300	10	139	8	1130	1.6			
29		39,500								
30		15,000								
31*										
32*										
33*										
34*										
35*										
36	17.5	12,500	6	388	75	618	1.06	618	0.82	
37	17.5	10,500	50	400	282	410	0.78	1180	1.48	
38	17.1	16,500	600	412	150	89.4	0.82	774	1.0	
39	17.1	7,000	60	296	88	590	1.17	1410	1.34	
40	17.7	15,000	600	200	372	956	2.43	1370	1.48	
41	17.6	15,000	6	400	120	488	1.0	976	1.04	
42	16.8	5,800	60	405	225	266	0.72	1330	1.40	
43	17.2	21,000	70	325	285	796	1.67	1500	1.72	
44	17.0	34,000	10	370	12	1000	1.36	1590	1.70	
45	17.2		4	30	0	1401	2.04	1500	1.58	
46	17.5	26,000	6	405	100	860	1.08	1590	1.72	
47	17.8	9,500	100	412	240	1095	1.30	1170	1.67	
48*										
49	17.3	12,500	29	200	112	1000	1.34	1410	1.44	
50	18.1	6,800	50	442	175	860	1.03	1775	1.92	
51	17.7	13,000	14	376	0	1050	1.47	2120	2.40	
52	17.7	6,200	2	335	150	1095	1.10	1230	1.42	
53		40,000		100	188	5660	5.2	2920	2.65	
54		13,500	400	425	202	1225	1.81	1685	2.10	
55		66,000	4.5	25	8	4820	5.2	5300	5.45	

* These exploratory formulations were evaluated subjectively and not characterized by quantitative tests.

(Continued)

TABLE XXIII (Continued)
EXPERIMENTAL FORMULATIONS PHYSICAL DATA

Formulation Number	Density (Wt/Gal)	Consistency @ 350°F (cps)	Creep Rate (in/in/min x 10 ⁻⁵)	Static Shear Bond		Flexural Impact @ 77°F		Flexural Impact @ 0°F		Vehicle Consistency @ 350°F (cps)
				@ 77°F (psi)	@ 0°F (psi)	Max. Fiber Stress (psi)	Absorbed Energy (in-lb/in ²)	Max. Fiber Stress (psi)	Absorbed Energy (in-lb/in ²)	
56		25,000	5		132	2820	2.94	4720	4.90	
57		1,100	0.6	112	75	391	1.12	548	1.50	
58			600		230	1370	2.96	905	1.96	50
59			15			2380	3.3			
60		60,000	5.5		0	4550	4.2	4550	4.70	12,000
61			62							900
62			9			1580	2.1	1940	2.16	
63		25,000	6		5	2310	3.1	2730	3.48	
64										
65			20			226	1.25			320
66			14.5			820	2.40			190
67			9			410	0.62			420
68			29							150
69			8			1000	1.3	1170	1.56	250
70			5			840	1.2	720	1.35	62
71										
72			34.2			900	1.32	1111	1.43	82
73			19	470	195	713	1.8	624	1.23	23
74						2240	2.9			8
75						4060	3.93			1300
76			0.2	35	0	1540	2.29			140
77				0	0					
78				202	17	725	0.84			42
79			2	15	10	975	1.16			210
80						7250	8.4			1500
81										3300
82			5	12	32	825	1.0			
83				480	300	825	1.7			

* These exploratory formulations were evaluated subjectively and not characterized by quantitative tests.

(Continued)

TABLE XXIII (Continued)
EXPERIMENTAL FORMULATIONS PHYSICAL DATA

Formulation Number	Density (Wt/Gal)	Consistency @ 350°F (cps)	Creep Rate (in/in/min $\times 10^{-3}$)	Static Shear Bond		Flexural Impact @ 77°F		Flexural Impact @ 0°F		Vehicle Consistency @ 350°F (cps)
				@ 77°F (psi)	@ 0°F (psi)	Max. Fiber Stress (psi)	Absorbed Energy (in-lb/in ²)	Max. Fiber Stress (psi)	Absorbed Energy (in-lb/in ²)	
84				110	25	825	1.0			
85										
86			16.0	380	95	1300	1.4			150
87										14
88			>600							
89			3.0	372	8	1200	1.5			
90			0.0			1560	1.5			50
91			5.0			1160	3.3			
92			5.5			1560	4.9			
93						2440	5.0			
94						1860	3.2			
95			92.0			9640	21.8			
96			0.2			7300	12.0			
97			0.4			5840	30.8			
98			0.2			8950	19.0			
99			0.4			5820	50.7			
100			0.3			7260	21.7			
101			>600							
102*										
103*										
104*										
105*										
106*										
107*										
108*										
109*										
110*										
111*										

*These exploratory formulations were evaluated subjectively and not characterized by quantitative tests.

(Continued)

TABLE XXIII (Continued)
EXPERIMENTAL FORMULATIONS PHYSICAL DATA

Formulation Number	Density (Wt/Gal)	Consistency @ 350°F (cps)	Creep Rate (in/in/min x 10 ⁻³)	Static Shear Bond		Flexural Impact @ 77°F		Flexural Impact @ 0°F		Vehicle Consistency @ 350°F (cps)
				@ 77°F (psi)	@ 0°F (psi)	Max. Fiber Stress (psi)	Absorbed Energy (in-lb/in ²)	Max. Fiber Stress (psi)	Absorbed Energy (in-lb/in ²)	
112*										
113										
114										14,000
115										5,200
116										
117										9,000
118										
119										6,000
120			>600			5125	96.6			2,400
121				278	500					2,200
122			14		500					
123						2800	8.0			4,000
124			0.6			391	1.1			1,100
125						3120	6.4			
126						2360	4.2			
127						3150	4.8			
128										1,700
129			160			6660	11.8			6,800
131			1.2	262	500		60.04			4,200
132										
132-1			Broke				60.04			
132-2			Too Soft							
132-3										
132-4			Too Soft							
132-5										
132-6			1.5			2270	10.5			
132-7			1.2			1590	8.3			

*These exploratory formulations were evaluated subjectively and not characterized by quantitative tests.

(Continued)

TABLE XXIII (Continued)
EXPERIMENTAL FORMULATIONS PHYSICAL DATA

Formulation Number	Density (Wt/Gal)	Consistency @ 350°F (cps)	Creep Rate (in/in/min x 10 ⁻³)	Static Shear Bond		Flexural Impact @ 77°F		Flexural Impact @ 0°F		Vehicle Consistency @ 350°F (cps)
				@ 77°F (psi)	@ 0°F (psi)	Max. Fiber Stress (psi)	Absorbed Energy (in-lb/in ²)	Max. Fiber Stress (psi)	Absorbed Energy (in-lb/in ²)	
132-8			2.0				60.04			
132-9			1.8			2360	7.0			
132-10			Broke			4625	114.6			
132-11			Cracked				60.04			
132-12			1.2			3358	42.3			
133										
133-1		Incompatible								
133-2		>10,000								
133-3			600			8980	56.2			7,100
133-4		Incompatible								
134			225	495	500	8525	9.4			
135			150			6439	11.4			2,200
136			150	472	420	9570	12.0			
137						3537	5.6			
138			82	89	6	5077	6.7			1,300
139										3,700
140			0.8			5850	171.5			
141						1500	67.0			
142										250
143			0.5			4800	6.4			
144			14			4860	83.1			
145						5310	20.2			
146										
147			4.0				60.0+			
148			58.0			3540	6.8			
149			81.0			1135	1.8			
150			32.0			1090	1.5			

(Continued)

TABLE XXIII (Concluded)
EXPERIMENTAL FORMULATIONS PHYSICAL DATA

Formulation Number	Density (Wt/Gal.)	Consistency @ 350°F (cps)	Creep Rate (in/in/min $\times 10^{-3}$)	Static Shear Bond		Flexural Impact @ 77°F		Flexural Impact @ 0°F		Vehicle Consistency @ 350°F (cps)
				@ 77°F (psi)	@ 0°F (psi)	Max. Fiber Stress (psi)	Absorbed Energy (in-lb/in ²)	Max. Fiber Stress (psi)	Absorbed Energy (in-lb/in ²)	
151			6.0			1044	1.2			
152			40.0			1044	1.0			
153			20.0			862	1.0			
154			10.0			1634	1.9			

FILE NO.		CONSTITUENTS DATA				MANUFACTURER	
RM NO.	CODE	PIG.CODE	COST				TRADE NAME
FORM NO.		WPG	LOT NO.				
GENERIC NAME		NVWP	QUAN.				
		NVWPG	DATE				
100151	MAGNESIUM SILICATE	GHP237100237	62	GOUN TALC	NYTAL	300	
100161	TITANIUM DIOXIDE RUTILE	TRP350100350	62	DUPONT TIO-610			
100171	DIATOMACEOUS SILICA	GFP192100192	62	JOHNS. MAN	CELITE	281	
100321	COBALT NAPHTHENATE	ND 080006095	171008220164	HARSHAW			
100331	LEAD NAPHTHENATE	ND 096067113	178008220164	HARSHAW			
100341	MANGANESE NAPHTHENATE	ND 081006098	139008220164	HARSHAW			
100351	TOLUENE	WT 072000000	036210164	NOTTINGHAM CO.			
100391	TRITOLYL PHOSPHATE	NG 082	290164	EASTMAN KODAK			
100441	MAGNESIUM SILICATE	GHP230100230	315	031049	DUPONT		
100451	MAGNESIUM SILICATE	GHP230100230			INTL TALC		
100581	ANTI-SKIN AGENT	NB 075000000			NUODEX		
100631	CAC03 FINE	GOP224100224	220864	GA. MARB. GAMACO			
100641	DIPENTENE	WX 072000000	030964	HERCULES NO. 122			
100661	ANTI-SKIN AGENT =2	NB 076000000	688	050964	NUODEX EXKIN NO.2		
100671	ANTI-SKIN AGENT =1	NB 076000000	381	050964	NUODEX EXKIN NO.1		
100691	PETROLEUM RESIN	HS	154	150964	PICCOFLEX 120		
100701	PETROLEUM RESIN	HS	034	150964	PICCOLASTIC E-75		
100721	LEAD PHOSPHITE	LXP678100678	372	290964	NATL. PB DYPHOS PG		
100731	AL<YD	AS	021064	RCI BECK P-670-55			
100741	CLAY THICKENER 38	NEP150100150	214001061064	NATL. PB			
100771	TOLUENE	WT 072000000	036231164	NOTTINGHAM CO.			
100781	GLYCERINE 99.50	NX	221264	DOW CHEM. VA ROSS			
100811	ESTER CUM	XX 089100	020190165	HERC. STAYBELITE 10			
100831	WHITE CAC03 GROUND	GDP 100	050201264	GA. MARB. 1= TATE			

FILE NO.		CONSTITUENTS		DATA		MANUFACTURER	
RM NO.	CODE	PIG.CODE	COST	LOT NO.	TRADE NAME		
FORM NO.		WPG		QUAN.			
GENERIC NAME		NVWP		DATE			
		NVWPG					
100841	WHITE CAC03 GROUND	GDP	100	050201264	GA. MARB. 2= TATE		
100851	CAC03 GROUND	GDP	100	100201264	GA. MARB. R101 40		
100881	BUTD. STYR. RESIN MILLED	JB 086		141050240265	GDYR. PLIOLITE ACL		
100891	HOT MELT			059100170365	PERMALINE		
100901	HOT MELT		100	178100180365	CATAPHOTE CATATHERM		
100921	SOLID ALKYD RESIN	AX		870001230365	RCI BECKASOL =23		
100931	EPOXIDE PLASTIC	EP		774001230365	UNION CARB. BAKELITE		
100941	2,4 DI-TERT-AMYLPHENOL	NI		290365	EASTMAN KODAK, WILL		
100951	BARYTES =1	GBP371100371		050	CK WILLIAMS		
100971	SILICA AMORPHOUS	GFP221100221		050020465	CK WILLIAMS		
100991	PETROLEUM RESIN	HS		050465	VELS. WYTON 2001		
101001	PETROLEUM RESIN	HS		050465	VELS. WYTON 4001		
101061	PHE. HG. AC. HG 180	NJ		0465	NUODEX PMA-18		
101071	HIGHWAY BEADS	GXP200100200		050			
101081	TIO RUTILE	TRP325100325		100280465	DUPONT TIO-610		
101091	ETHYL CELLULOSE	CX 100		114100300465	HERC.		
101101	ESTER GUM	XX 089		100060565	HERC. STAYB. ESTER10		
101111	GLASS BEADS	GXP200100200		120565	CATAPHORE CORP.		
101121	RUBBER ADHESIVE	JB		120465	GDYR. PLIOGRIP ADS74		
101141	MALEIC RESIN	IX 100		050200565	HERC. PENTALYN-K		
101161	CASTOR OIL	NK 080100080		210465	BAKER CASTOR OIL =15		
101171	PETROLEUM RESIN	HS		240565	VELS. STIKVEL W		
101221	TOLUENE	WT 072000000		100665	NOTTINGHAM CO.		
101231	XYLENE	WZ 072000000		100665	NOTTINGHAM CO.		
101281	CLAY THICKENER 27	NEP150100150		050865	NATL. PB BENTONE 27		

FILE NO.		CONSTITUENTS DATA				MANUFACTURER	
RM NO.	CODE	PIG.CODE	COST	LOT NO.	TRADE NAME		
FORM NO.		WPG	NVWP	QUAN.			
GENERIC NAME		NVWPG		DATE			
101291CLAY THICKENER 34		NEP200100200		050865	NATL. PB BENTONE 34		
101301CLAY THICKENER 38		NEP150100150		050865	NATL. PB BENTONE 38		
101391GLID.TRAFF.VEH.ALK YD		AS	209	170865	GLIDD.CO.		
101421HYDOX.ETHYLCELLULOSE		CX	100		UNION CARB.		
101461ANTI FOAM AGENT		NF	916		ADV.DIV		
101471ANTI FOAM AGNET		NF	000		ADV.DIV		
101501EPICHLOROHYDRIN		IX	100		EAST.CHEM.CO.		
101511CHLOR.POLYPHENYL		XR	100	R26	MONSANTO AROCHL-1254		
101521CHLOR.RUBBER10CPS		XR	136100	486	0565HERCULES PARLON SIO		
101531CHLOR.RUBBER 6CPS		XR	136100	446	0565HERCULES PARION S-6		
101541CHLOR.RUBBER20CPS		XR	136100		HERCULES PARLON S-20		
101551SOL G LOW BP AROMATIC SOL		WJ	070000		AM.MIN.SPIRITS SOLVG		
101571CALCIUM CARBONATE		GDP224100		140965	GEORGIA MARB\$ R040		
101581CALCIUM CARBONATE		GDP224100			GEORGIA MARB. 3050		
101591CALCIUM CARBONATE		GDP224100			GEORGIA MARB. X0 WH		
101601CALCIUM CARBONATE		GDP224100			GEORGIA MARB. P4-40		
101611ALKYD VEHICLE P-670-55		AS	076	140965	RCI BECKOSOL		
101621HOT MELT PROP. A			100	572	150965CATAPHOTE CATATHERM		
101641HOT MELT PROP. B			100	270965	PERMALINE 4F65BS9		
101691ASBESTOES SHORTS		YXP230100		7\$5	JOH.MANS.		
101701SANITARY CANS					SEARS ROEBUCK		
101711GLASS BEADS		YXP200100		251065	3M CO. SMALL TYPE		
101721GLASS BEADS		YXP200100		251065	CATAPHOTE		
10173140PERCENT PHTH.NONDRY		AS	100	136	NELIO RESIN VBR5010		
101741ALKYD NONDRY		AS	093100	241165	UNION CAMP VBR5010		

FILE NO.		CONSTITUENTS DATA					
RM NO.	CODE	PIG.CODE	COST	MANUFACTURER			TRADE NAME
FORM NO.		WPG	LOT NO.	QUAN.	DATE		
GENERIC NAME		NVWP					
		NVWPG					
101751	ALKYD NONDRY HARD MALEIC	AS 093100	114			UNION CAMP	VBR-701
101761	POLYPROPYLENE GUM M-5	HA 093100				EAST.CHEM	EASTOBOND
101771	ALKYD NONDRY	AS 093100	124		301165	UNION CAMP	VBR204
101781	ALKYD NONDRY	AS 093100	054		301165	UNION CAMP	VBR201
101791	ALKYD NONDRY	AS 093100	026		301165	UNION CAMP	VBR7022
101801	ALKYD NONDRY	AS 093100	196		011265	UNION CAMP	VBR5010
101811	ALKYD NONDRY	AS 093100	919		141265	UNION CAMP	VBR5010
101821	WETTING AGENT	NZ 081	707		171265	ADNANCE	ADVAVET100
101831	CALCIUM CARBONATE	GDP224100			171265	GA.MARB.	XO
101841	CALCIUM CARBONATE	GDP224100				GA MARB	BO
101851	STR.TALL OIL ROSIN	HX 090100	036			GLIDDEN	CHEM. S-80
101881	SMALL GLASS BEADS	YXP200100			663M		SIZE103-5005
101891	TALL OIL ROSIN 88PERCENT	HX 090100			660NION	CAMP	UNITOL 5
101911	SAIB	CX			66EASTMAN		
101921	EPOXY RESIN	EX 100			66SHELL	EPON1001	
101931	CELL.AC.BUTYRATE 1/2 SEC	CB 114100	507		180266	EASTMAN	EAB-550-1
101941	CELL.AC.BUTYRATE	CB 120100			180266	EASTMAN	EAB-500-1
101951	SLAKED LIME	YXP190100			66WILL	CORP	
101961	POLYMER HOTMELT STABCH201	NI 090100			250266	ADVANCE	ADVASTAB
101971	HYDROGENATED ROSIN	HX 090100			150465	HERCULES	ABITOL
101981	METAL RESINATE	HX 096100			250266	TENNCO	NIREZ V-2040
101991	POLYESTER LOW MW	XX 120100			030366	GOODYEAR	VITELP207
102001	POLYESTER	XX 120100			030366	GOODYEAR	VITELP200
102011	ESTER GUM	HX 090100			080366	HERCULES	STAYBELIT10

CONSTITUENTS DATA						
FILE NO.	CODE	PIG.CODE	COST	MANUFACTURER		
RM NO.		WPG	LOT NO.	TRADE NAME		
FORM NO.		NVWP	QUAN.			
GENERIC NAME		NVWPG	DATE			
102021HYDROGENATED ROSIN		HX 090100	030366	HERCULES	ABITOL	
102031POLYESTER		XX 120100	030366	GOODYEAR	PE207VITEL	
102041CHLOR.POLYPHENYLS		NK 100100	220466	MONSANTO	AROCL.5460	
102051ALKYD NONDRY		AS 090100	170565	UNION CAMP	VBR5010	
102061POLYESTER		XX 120100	280366	GOODYEAR	VITELPE200	
102071POLYESTER		XX 120100	280366	GOODYEAR	VITELPE207	
102081POLYAMIDE		XX 080100	300366	LAWTER	POLYMID1560	
102091POLYAMIDE		XX 080100	310366	GEN.MILLS	VER.1175	
102101CA CARBONATE		GDP224100	080466	GA.MARB.	RO 40	
102111CA CARBONATE		GDP224100	080466	GA.MARB.	XO	
102121PLASTIZER		NK 090100	130466	MONSANTO	SANT.NO.8	
102141TRIISOCTYL PHOSPHITE		NG 098100	116	180466	V-C CHEM.	
102151WAX MICROCRYSTAL.		NH 070100		HOECHST	TYPE E	
102161ZINC RESINATE		HX 100100	856	190466	NEWPORT ZIREX	
102171POLYAMIDE		XX 080100		270466	LAWTER POLYMIDE1560	
102181CA CARBONATE		GDP224100		180566	GA.MARB. RO-40	
102191CA CARBONATE		GDP224100		180566	GA.MARB. RO-40	
102201POLYAMIDE		XX 080100	253	190566	LAWTER POLYMIDE1560	
102211SMALL GLASS BEADS		YXP200100		170566	OM SIZE103-5005	
102221PLASTIZER		NK 090100		190466	MONSANTO SANTIZCER 8	
102231HOT MELT PROP. C		250100	587	190466	CATAPHOTE CATATHERM	
102241ALIPH. SOLVENT		WI 062			ROSE LACTOL	
102261EPOXY CURING AGT.		EX 090100			MAG PLAST. V-25	
102271ACETONE		WO 066			CHEM.SER.	

FILE NO.		CONSTITUENTS		DATA		MANUFACTURER	
RM NO.	CODE	PIG.CODE	COST	LOT NO.	TRADE NAME		
FORM NO.		WPG	NVWP	QUAN.			
GENERIC NAME			NVWPG	DATE			
102281GLASS BEADS REG.		YXP200100		020866	IND.GLASS.		
102481ALKYD L143-100		AS080		100767	REICH. BECKSOLIO-980		
102491ALKYD RESIN		AS0891100		100767	REICH. BECKSITE43102		
102671PETROLEUM RESIN		HS095100		1LB150964	PENN.IND.PICCOTEX100		
102681PETROLEUM RESIN		HS095100		1LB150964	PENN IND PICCOPALEH2		
102691ETHYL CELLULOSE		CX097100		1LB000065	HER. ETHOCEL N505		
102701CASTOR OIL NO.3		OX 080100		2LB000066	BAKER CASTOR OIL CO.		
102711TEMP.STABILIZER		IX		1LB030465	ADVA. ERL 277-4		
102721PENTYL PHENOL		IX		2LB	66EASTMAN KODAK		
102731POLYURETHANE RUBBER		UX 105100		5LB	64GOODYEAR ESTANE		
102741EPCXY RESIN		EP 095100			66SHELL CHEM.EPON 1007		
102751POLYAMIDE		XX 082100			66GEN.MILL VERS. 900		
102761SULFUR		IX 172100			64GULF		
102771POLYCARBONATE RESIN		XX 100100		5LB	65GE LEXAN		
102781BUTYL PHENOL		IX 081			65EASTMAN KODAK		
102791POLYESTER		XX			65GEN.MILLCX1012		
102801ME.PHTH.ET.GLYCOLLATE		IX 101100			66MONSANTO SANT. M-17		
102811PHOSPHORIC ACID		IX 141			66FISHER SCI.		
102821DIBUTYL PHTHALATE		IX 087100			66UNION CARBIDE		
102831CA ACETATE		IX			66FISHER SCI.		
102841EPOXY RESIN		EP 095			66SHELL EPON 1004		
102851FIBERGLASS CHOPPED MED.		RX 216100			66OWENS CORNING		
102861FIBERGLASS CHOPPED FINE		RX 216100			66OWENS CORNING		
102871SAND		RX 216			66SWANEE SAND CO.		

FILE NO.		CODE	CONSTITUENTS		DATA		MANUFACTURER	TRADE NAME
RM NO.	FORM NO.		PIG.CODE	WPG	COST	LOT NO.		
GENERIC NAME				NVWP	NVWPG	QUAN.	DATE	
102881	EPOXY RESIN		EP 095100				65SHELL CHEM EPON 1007	
102891	ALKYD RESIN		AM 080		089		66ADM TR-266-1	
102901	ALIPHATIC SOLVENT		WI063			5G.	65AMSCO LACTOL	
102911	EPOXY RESIN		EP090			1G.	66SHELL EPON 828	
102921	CURING AGENT		XX080			1G.	66SHELL V-25 CUR.AGT.	
102931	ACETONE		W0066			5G.	66CHEM.SER.	
102941	PLASTIZER		NH102			1LB	66MONSANTO SANT. 140	
102951	PLASTIZER		NH102			1LB	66MONSANTO SANT. 141	
102961	PLASTIZER		NH102			1LB	66MONSANTO SANT. 160	
102971	ARYLSULFONAMIDE FORM.RESIN	XX112				1LB	66MONSANTO SANT.MHP	
102981	PETROLEUM RESIN		HS090			1LB	64PENN IND.PICCO M-25	
102991	PARAFFIN WAX		HS083			1LB	64GULF	
103001	ROSIN DERIVATIVE		Q 085			1LB	64HERC. CELLOLYN 95	

ANALYSIS OF VARIANCE

SERIES I

Integrity Only
Concrete Only
Exposure 12 Months

Letter	Independent Variables	Variable Identification	
I	Formulation	1 Proprietary B #108 2 Proprietary A #109 3 $\frac{1}{2}$ S/A #16 4 1 S/A #15	5 2x S/A #19 6 Hydrocarbon #21 7 Ethyl Cellulose #24 8 Alkyd #22
J	Thickness	1 $\frac{3}{32}$ " 2 $\frac{3}{16}$ "	
K	Priming	1 None 2 Rubber 3 Epoxy	

Computation of Confidence Limits

$$S_{\bar{x}} = \sqrt{\frac{S-2}{N}} ; 95\% \text{ Confidence Range} = \bar{x} \pm t S_{\bar{x}}$$

	$\frac{S-2}{N}$	N	DF	t	$\frac{S-2}{N}$	$\frac{S-2}{N}$	95% C.R.
Formulation	0.639	6	14	2.14	.106	.326	.675
Thickness	0.639	24	14	2.14	.026	.162	.346
Priming	0.639	16	14	2.14	.040	.20	.427
Formulation (no primer)	0.845	2	7	2.36	.422	.650	1.53
Thickness (no primer)	0.845	8	7	2.36	.054	.232	0.55

ANALYSIS OF VARIANCE

SOURCE	DF	SS	MS	F
TOTAL	47	1.43916666665e+02		
IJK	14	8.95833332840e+00	6.39880952030e-01	
IJ	7	5.91666667160e+00	8.45238095950e-01	
IJK	14	8.95833332840e+00	6.39880952030e-01	1.32093023440e+00
IK	14	1.97916666717e+01	1.41369047655e+00	
IJK	14	8.95833332840e+00	6.39880952030e-01	2.20930232736e+00 *
JK	2	5.04166667163e+00	2.52083333582e+00	
IJK	14	8.95833332840e+00	6.39880952030e-01	3.93953488978e+00 **
I	7	5.85833333280e+01	8.36904761840e+00	
IJK	14	8.95833332840e+00	6.39880952030e-01	1.30790697735e+01 ***
J	1	1.00833333284e+01	1.00833333284e+01	
IJK	14	8.95833332840e+00	6.39880952030e-01	1.57581395358e+01 ***
K	2	3.55416666642e+01	1.77708333320e+01	
IJK	14	8.95833332840e+00	6.39880952030e-01	2.77720930366e+01 ***

*SIGNIFICANT AT 10%

**SIGNIFICANT AT 5%

***SIGNIFICANT AT 1%

I	J	K	MEAN
0	0	0	7.291666666660E+00
0	0	1	6.562500000000E+00
0	0	2	6.812500000000E+00
0	0	3	8.500000000000E+00
0	1	0	6.833333333330E+00
0	1	1	5.875000000000E+00
0	1	2	6.125000000000E+00
0	1	3	8.500000000000E+00
0	2	0	7.750000000000E+00
0	2	1	7.250000000000E+00
0	2	2	7.500000000000E+00
0	2	3	8.500000000000E+00
1	0	0	7.000000000000E+00
1	0	1	5.500000000000E+00
1	0	2	6.500000000000E+00
1	0	3	9.000000000000E+00
1	1	0	6.000000000000E+00
1	1	1	4.000000000000E+00
1	1	2	5.000000000000E+00
1	1	3	9.000000000000E+00
1	2	0	8.000000000000E+00
1	2	1	7.000000000000E+00
1	2	2	8.000000000000E+00
1	2	3	9.000000000000E+00
2	0	0	7.000000000000E+00
2	0	1	7.000000000000E+00
2	0	2	6.500000000000E+00
2	0	3	7.500000000000E+00
2	1	0	6.000000000000E+00
2	1	1	6.000000000000E+00
2	1	2	5.000000000000E+00
2	1	3	7.000000000000E+00
2	2	0	8.000000000000E+00
2	2	1	8.000000000000E+00
2	2	2	8.000000000000E+00
2	2	3	8.000000000000E+00
3	0	0	8.166666666660E+00
3	0	1	7.500000000000E+00
3	0	2	7.500000000000E+00
3	0	3	9.500000000000E+00
3	1	0	7.666666666670E+00
3	1	1	7.000000000000E+00
3	1	2	7.000000000000E+00
3	1	3	9.000000000000E+00
3	2	0	8.666666666660E+00
3	2	1	8.000000000000E+00
3	2	2	8.000000000000E+00
3	2	3	1.000000000000E+01
4	0	0	7.500000000000E+00
4	0	1	6.000000000000E+00
4	0	2	6.500000000000E+00
4	0	3	1.000000000000E+01
4	1	0	7.333333333330E+00
4	1	1	6.000000000000E+00
4	1	2	6.000000000000E+00
4	1	3	1.000000000000E+01
4	2	0	7.666666666670E+00
4	2	1	6.000000000000E+00
4	2	2	7.000000000000E+00
4	2	3	1.000000000000E+01

5	0	0	4.666666666666e+00
5	0	1	3.999999999999e+00
5	0	2	3.999999999999e+00
5	0	3	6.000000000000e+00
5	1	0	4.666666666667e+00
5	1	1	4.000000000000e+00
5	1	2	4.000000000000e+00
5	1	3	6.000000000000e+00
5	2	0	4.666666666667e+00
5	2	1	4.000000000000e+00
5	2	2	4.000000000000e+00
5	2	3	6.000000000000e+00
6	0	0	7.500000000000e+00
6	0	1	7.500000000000e+00
6	0	2	7.000000000000e+00
6	0	3	8.000000000000e+00
6	1	0	7.333333333330e+00
6	1	1	7.000000000000e+00
6	1	2	7.000000000000e+00
6	1	3	8.000000000000e+00
6	2	0	7.666666666670e+00
6	2	1	8.000000000000e+00
6	2	2	7.000000000000e+00
6	2	3	8.000000000000e+00
7	0	0	8.166666666660e+00
7	0	1	6.500000000000e+00
7	0	2	9.000000000000e+00
7	0	3	9.000000000000e+00
7	1	0	7.666666666670e+00
7	1	1	5.000000000000e+00
7	1	2	9.000000000000e+00
7	1	3	9.000000000000e+00
7	2	0	8.666666666660e+00
7	2	1	8.000000000000e+00
7	2	2	9.000000000000e+00
7	2	3	9.000000000000e+00
8	0	0	8.333333333330e+00
8	0	1	8.500000000000e+00
8	0	2	7.500000000000e+00
8	0	3	9.000000000000e+00
8	1	0	8.000000000000e+00
8	1	1	8.000000000000e+00
8	1	2	6.000000000000e+00
8	1	3	1.000000000000e+01
8	2	0	8.666666666660e+00
8	2	1	9.000000000000e+00
8	2	2	9.000000000000e+00
8	2	3	8.000000000000e+00

ANALYSIS OF VARIANCE

SERIES I

Integrity Only
Concrete Only
Exposure 24 Months

<u>Letter</u>	<u>Independent Variables</u>	<u>Variable Identification</u>	
I	Formulation	1 Proprietary B #108 2 Proprietary A #109 3 $\frac{1}{2}$ S/A #16 4 1 S/A #15	5 2x S/A #19 6 Hydrocarbon #21 7 Ethyl Cellulose #24 8 Alkyd #22
J	Thickness	1 $\frac{3}{32}$ " 2 $\frac{3}{16}$ "	
K	Priming	1 None 2 Rubber 3 Epoxy	

Computation of Confidence Limits

$$S_{\bar{x}} = \sqrt{\frac{S^2}{N}} ; 95\% \text{ Confidence Range} = \bar{x} \pm tS_{\bar{x}}$$

	$\frac{S^2}{x}$	<u>N</u>	<u>DF</u>	<u>t</u>	$\frac{S^2}{x}$ <u>N</u>	$\frac{S}{x}$	$tS_{\bar{x}}$
Formulation	1.07	6	14	2.14	.178	.423	.904
Thickness	1.07	21	14	2.14	.051	.226	.483
Priming	1.07	14	14	2.14	.076	.277	.593

ANALYSIS OF VARIANCE

SOURCE	DF	SS	MS	F
TOTAL	47	1.97666666664e+02		
IJK	14	1.49999999926e+01	1.07142857090e+00	
IJ	7	5.25000000745e+00	7.50000001060e-01	
IJK	14	1.49999999926e+01	1.07142857090e+00	7.00000001340e-01
IK	14	3.36666666716e+01	2.40476190511e+00	
IJK	14	1.49999999926e+01	1.07142857090e+00	2.24444444588e+00 *
JK	2	4.66666667163e+00	2.33333333582e+00	
IJK	14	1.49999999926e+01	1.07142857090e+00	2.17777778118e+00
I	7	7.43333333280e+01	1.06190476184e+01	
IJK	14	1.49999999926e+01	1.07142857090e+00	9.91111111540e+00 ***
J	1	1.40833333283e+01	1.40833333283e+01	
IJK	14	1.49999999926e+01	1.07142857090e+00	1.31444444464e+01 ***
K	2	5.06666666642e+01	2.53333333321e+01	
IJK	14	1.49999999926e+01	1.07142857090e+00	2.36444444550e+01 ***

**SIGNIFICANT AT 5%

***SIGNIFICANT AT 1%

I	J	K	MEAN
0	0	0	6.41666666666600E+00
0	0	1	5.24999999999900E+00
0	0	2	6.25000000000000E+00
0	0	3	7.75000000000000E+00
0	1	0	5.87500000000000E+00
0	1	1	4.37500000000000E+00
0	1	2	5.62500000000000E+00
0	1	3	7.62500000000000E+00
0	2	0	6.95833333333300E+00
0	2	1	6.12500000000000E+00
0	2	2	6.87500000000000E+00
0	2	3	7.87500000000000E+00
1	0	0	5.16666666666600E+00
1	0	1	2.99999999999900E+00
1	0	2	4.99999999999900E+00
1	0	3	7.50000000000000E+00
1	1	0	4.33333333333300E+00
1	1	1	3.00000000000000E+00
1	1	2	3.00000000000000E+00
1	1	3	7.00000000000000E+00
1	2	0	6.00000000000000E+00
1	2	1	3.00000000000000E+00
1	2	2	7.00000000000000E+00
1	2	3	8.00000000000000E+00
2	0	0	7.00000000000000E+00
2	0	1	5.50000000000000E+00
2	0	2	7.50000000000000E+00
2	0	3	8.00000000000000E+00
2	1	0	6.33333333333300E+00
2	1	1	4.00000000000000E+00
2	1	2	7.00000000000000E+00
2	1	3	8.00000000000000E+00
2	2	0	7.66666666666700E+00
2	2	1	7.00000000000000E+00
2	2	2	8.00000000000000E+00
2	2	3	8.00000000000000E+00
3	0	0	7.83333333333300E+00
3	0	1	7.00000000000000E+00
3	0	2	7.00000000000000E+00
3	0	3	9.50000000000000E+00
3	1	0	7.00000000000000E+00
3	1	1	6.00000000000000E+00
3	1	2	6.00000000000000E+00
3	1	3	9.00000000000000E+00
3	2	0	8.66666666666600E+00
3	2	1	8.00000000000000E+00
3	2	2	8.00000000000000E+00
3	2	3	1.00000000000000E+01
4	0	0	6.33333333333300E+00
4	0	1	5.50000000000000E+00
4	0	2	5.50000000000000E+00
4	0	3	8.00000000000000E+00
4	1	0	6.33333333333300E+00
4	1	1	6.00000000000000E+00
4	1	2	5.00000000000000E+00
4	1	3	8.00000000000000E+00
4	2	0	6.33333333333300E+00
4	2	1	5.00000000000000E+00
4	2	2	6.00000000000000E+00
4	2	3	8.00000000000000E+00

5	0	0	4.166666666666666E+00
5	0	1	3.499999999999999E+00
5	0	2	3.999999999999999E+00
5	0	3	4.999999999999999E+00
5	1	0	4.000000000000000E+00
5	1	1	3.000000000000000E+00
5	1	2	4.000000000000000E+00
5	1	3	5.000000000000000E+00
5	2	0	4.333333333333333E+00
5	2	1	4.000000000000000E+00
5	2	2	4.000000000000000E+00
5	2	3	5.000000000000000E+00
6	0	0	5.666666666666666E+00
6	0	1	5.500000000000000E+00
6	0	2	4.999999999999999E+00
6	0	3	6.500000000000000E+00
6	1	0	5.333333333333333E+00
6	1	1	5.000000000000000E+00
6	1	2	5.000000000000000E+00
6	1	3	6.000000000000000E+00
6	2	0	6.000000000000000E+00
6	2	1	6.000000000000000E+00
6	2	2	5.000000000000000E+00
6	2	3	7.000000000000000E+00
7	0	0	7.333333333333333E+00
7	0	1	4.499999999999999E+00
7	0	2	9.500000000000000E+00
7	0	3	8.000000000000000E+00
7	1	0	6.333333333333333E+00
7	1	1	2.000000000000000E+00
7	1	2	9.000000000000000E+00
7	1	3	8.000000000000000E+00
7	2	0	8.333333333333333E+00
7	2	1	7.000000000000000E+00
7	2	2	1.000000000000000E+01
7	2	3	8.000000000000000E+00
8	0	0	7.833333333333333E+00
8	0	1	7.500000000000000E+00
8	0	2	6.500000000000000E+00
8	0	3	9.500000000000000E+00
8	1	0	7.333333333333333E+00
8	1	1	6.000000000000000E+00
8	1	2	6.000000000000000E+00
8	1	3	1.000000000000000E+01
8	2	0	8.333333333333333E+00
8	2	1	9.000000000000000E+00
8	2	2	7.000000000000000E+00
8	2	3	9.000000000000000E+00

ANALYSIS OF VARIANCE

SERIES I

Integrity Only
Concrete and Asphalt
Exposure 12 Months

<u>Letter</u>	<u>Independent Variable</u>	<u>Variable Identification</u>	
I	Substrate	1 Concrete 2 Asphalt	
J	Formulation	1 Proprietary B #108 2 Proprietary A #109 3 $\frac{1}{2}$ S/A #16 4 1 S/A #15	5 2x S/A #19 6 Hydrocarbon #21 7 Ethyl Cellulose #24 8 Alkyd #22
K	Thickness	1 3/32" 2 3/16"	

Computation of Confidence Limit

$$S_{\bar{x}} = \sqrt{\frac{S^2}{N}} ; 95\% \text{ Confidence Range} = \bar{x} \pm tS_{\bar{x}}$$

	$\frac{S^2}{x}$	<u>N</u>	<u>DF</u>	<u>t</u>	$\frac{S^2}{x}$ <u>N</u>	$\frac{S}{x}$	$tS_{\bar{x}}$
Substrate	3.07	16	7	2.36	.192	.438	1.03
Formulation	3.07	4	7	2.36	.765	.875	2.07
Thickness	3.07	16	7	2.36	.192	.438	1.03

ANALYSIS OF VARIANCE

SOURCE	DF	SS	MS
TOTAL	31	1.158750000000e+02	
IJK	7	2.149999999999e+01	3.07142857143e+00
IJ	7	2.850000000001e+01	4.07142857143e+00
IJK	7	2.149999999999e+01	3.07142857143e+00 1.32558139534e+00
IK	1	1.250000000000e+01	1.250000000000e+01
IJK	7	2.149999999999e+01	3.07142857143e+00 4.06976744185e+00 *
JK	7	3.087499999999e+01	4.41071428571e+00
IJK	7	2.149999999999e+01	3.07142857143e+00 1.43604651162e+00
I	1	5.000000000000e-01	5.000000000000e-01
IJK	7	2.149999999999e+01	3.07142857143e+00 1.62790697673e-01
J	7	2.187500000000e+01	3.125000000000e+00
IJK	7	2.149999999999e+01	3.07142857143e+00 1.01744186046e+00
K	1	1.250000000000e-01	1.250000000000e-01
IJK	7	2.149999999999e+01	3.07142857143e+00 4.06976744185e-02

*SIGNIFICANT AT 10% LEVEL

I	J	K	MEAN
0	0	0	6.717500000000e+00
0	0	1	6.375000000000e+00
0	0	2	6.500000000000e+00
0	1	0	7.000000000000e+00
0	1	1	6.000000000000e+00
0	1	2	8.000000000000e+00
0	2	0	6.750000000000e+00
0	2	1	6.000000000000e+00
0	2	2	7.500000000000e+00
0	3	0	7.500000000000e+00
0	3	1	7.500000000000e+00
0	3	2	7.500000000000e+00
0	4	0	6.250000000000e+00
0	4	1	6.500000000000e+00
0	4	2	6.000000000000e+00
0	5	0	4.500000000000e+00
0	5	1	4.500000000000e+00
0	5	2	4.500000000000e+00
0	6	0	6.250000000000e+00
0	6	1	6.000000000000e+00
0	6	2	6.500000000000e+00
0	7	0	6.500000000000e+00
0	7	1	5.500000000000e+00
0	7	2	7.500000000000e+00
0	8	0	6.750000000000e+00
0	8	1	9.000000000000e+00
0	8	2	4.500000000000e+00
1	0	0	6.562500000000e+00
1	0	1	5.875000000000e+00
1	0	2	7.250000000000e+00
1	1	0	5.500000000000e+00
1	1	1	4.000000000000e+00
1	1	2	7.000000000000e+00
1	2	0	7.000000000000e+00
1	2	1	6.000000000000e+00
1	2	2	8.000000000000e+00
1	3	0	7.500000000000e+00
1	3	1	7.000000000000e+00
1	3	2	8.000000000000e+00
1	4	0	6.000000000000e+00
1	4	1	6.000000000000e+00
1	4	2	6.000000000000e+00
1	5	0	4.000000000000e+00
1	5	1	4.000000000000e+00
1	5	2	4.000000000000e+00
1	6	0	7.500000000000e+00
1	6	1	7.000000000000e+00
1	6	2	8.000000000000e+00
1	7	0	6.500000000000e+00
1	7	1	5.000000000000e+00
1	7	2	8.000000000000e+00
1	8	0	8.500000000000e+00
1	8	1	8.000000000000e+00
1	8	2	9.000000000000e+00
2	0	0	6.312500000000e+00
2	0	1	6.875000000000e+00
2	0	2	5.750000000000e+00
2	1	0	8.500000000000e+00
2	1	1	8.000000000000e+00
2	1	2	9.000000000000e+00

2	2	0	6.500000000000E+00
2	2	1	6.000000000000E+00
2	2	2	7.000000000000E+00
2	3	0	7.500000000000E+00
2	3	1	8.000000000000E+00
2	3	2	7.000000000000E+00
2	4	0	6.500000000000E+00
2	4	1	7.000000000000E+00
2	4	2	6.000000000000E+00
2	5	0	5.000000000000E+00
2	5	1	5.000000000000E+00
2	5	2	5.000000000000E+00
2	6	0	5.000000000000E+00
2	6	1	5.000000000000E+00
2	6	2	5.000000000000E+00
2	7	0	6.500000000000E+00
2	7	1	6.000000000000E+00
2	7	2	7.000000000000E+00
2	8	0	7.000000000000E+00
2	8	1	1.000000000000E+01
2	8	2	9.000000000000E+00

ANALYSIS OF VARIANCE

SERIES I

Night Visibility Only
Concrete and Asphalt
Exposure 12 Months

Letter	Independent Variables	Variable Identification	
I	Substrate	1 Concrete 2 Asphalt	
J	Formulation	1 Proprietary B #108 2 Proprietary A #109 3 $\frac{1}{2}$ S/A #16 4 1 S/A #15	5 2x S/A #19 6 Hydrocarbon #21 7 Ethyl Cellulose #24 8 Alkyd #22

Computation of Confidence Limits

$$S_{\bar{x}} = \sqrt{\frac{S^2}{N}} ; 95\% \text{ Confidence Range} = \bar{x} \pm tS_{\bar{x}}$$

	$\frac{S^2}{x}$	\underline{N}	\underline{DF}	\underline{t}	$\frac{S^2}{x}$ \underline{N}	$\underline{S_{\bar{x}}}$	$\underline{tS_{\bar{x}}}$
Substrate	.964	8	7	2.36	.120	.347	.817
Formulation	.964	2	7	2.36	.482	.695	1.64

ANALYSIS OF VARIANCE

SOURCE	DF	SS	MS	F
TOTAL	15	9.975000000000e+01		
INTER	7	6.750000000000e+00	9.64285714280e-01	
I	1	2.250000000000e+00	2.250000000000e+00	
INTER	7	6.750000000000e+00	9.64285714280e-01	2.33333333333e+00
J	7	9.075000000000e+01	1.29642857143e+01	
INTER	7	6.750000000000e+00	9.64285714280e-01	1.34444444445e+01 ***

***SIGNIFICANT AT 1% LEVEL

at 1% level

I	J	MEAN
0	0	8.625000000000E+00
0	1	1.050000000000E+01
0	2	6.500000000000E+00
0	3	1.149999999999E+01
0	4	1.100000000001E+01
0	5	4.500000000000E+00
0	6	6.500000000000E+00
0	7	1.000000000000E+01
0	8	8.500000000000E+00
1	0	9.000000000000E+00
1	1	1.000000000000E+01
1	2	8.000000000000E+00
1	3	1.100000000001E+01
1	4	1.200000000000E+01
1	5	5.000000000000E+00
1	6	7.000000000000E+00
1	7	1.000000000000E+01
1	8	9.000000000000E+00
2	0	8.250000000000E+00
2	1	1.100000000001E+01
2	2	5.000000000000E+00
2	3	1.200000000000E+01
2	4	1.000000000000E+01
2	5	4.000000000000E+00
2	6	6.000000000000E+00
2	7	1.000000000000E+01
2	8	8.000000000000E+00

ANALYSIS OF VARIANCE

SERIES I

Integrity Only
Asphalt Only
Exposure 12 Months

<u>Letter</u>	<u>Independent Variable</u>	<u>Variable Identification</u>	
I	Formulation	1 Proprietary B #108	5 2x S/A #19
		2 Proprietary A #109	6 Hydrocarbon #21
		3 1/2 S/A #16	7 Ethyl Cellulose #24
		4 1 S/A #15	8 Alkyd #22
J	Thickness	1 3/32"	
		2 3/16"	

Computation of Confidence Limit

$$S_{\frac{x}{x}} = \sqrt{\frac{S-\frac{2}{x}}{N}} ; 95\% \text{ Confidency Range} = \bar{x} \pm tS_{\frac{x}{x}}$$

	<u>$\frac{S-\frac{2}{x}}{x}$</u>	<u>N</u>	<u>DF</u>	<u>t</u>	<u>$\frac{S-\frac{2}{x}}{N}$</u>	<u>$\frac{S-\frac{2}{x}}{N}$</u>	<u>$tS_{\frac{x}{x}}$</u>
Formulation	6.77	2	7	2.36	3.38	1.84	4.34
Thickness	6.77	8	7	2.36	.845	.022	2.17

ANALYSIS OF VARIANCE

SOURCE	DF	SS	MS	F
TOTAL	15	7.543750000000+01		
INTER	7	4.743749999999+01	6.776785714300+00	
I	7	2.293750000000+01	3.276785714290+00	
INTER	7	4.743749999999+01	6.776785714300+00	4.835309617920-01
J	1	5.062500000000+00	5.062500000000+00	
INTER	7	4.743749999999+01	6.776785714300+00	7.470355731200-01
NO SIGNIFICANT FACTORS				

I	J	MEAN
0	0	6.312500000000e+00
0	1	6.875000000000e+00
0	2	6.870000000000e+00
1	0	8.500000000000e+00
1	1	8.000000000000e+00
1	2	9.000000000000e+00
2	0	6.500000000000e+00
2	1	6.000000000000e+00
2	2	7.000000000000e+00
3	0	7.500000000000e+00
3	1	8.000000000000e+00
3	2	7.000000000000e+00
4	0	6.500000000000e+00
4	1	7.000000000000e+00
4	2	6.000000000000e+00
5	0	5.000000000000e+00
5	1	5.000000000000e+00
5	2	5.000000000000e+00
6	0	5.000000000000e+00
6	1	5.000000000000e+00
6	2	5.000000000000e+00
7	0	6.500000000000e+00
7	1	6.000000000000e+00
7	2	7.000000000000e+00
8	0	5.000000000000e+00
8	1	1.000000000000e+01
8	2	9.000000000000e+00

ANALYSIS OF VARIANCE

SERIES II

Integrity Only
Concrete Only
Exposure 12 Months

<u>Letter</u>	<u>Independent Variable</u>	<u>Variable Identification</u>	
I	Formulation	1 Polyamide #155	5 Alkyd #162
		2 Polyamide #157	6 Alkyd #163
		3 Polyamide #158	7 Proprietary #164
		4 1 S/A #160	
J	Priming	1 None	
		2 Epoxy	

Computation of Confidence Limits

$$S_{\bar{x}} = \sqrt{\frac{S_x^2}{N}} ; 95\% \text{ Confidence Range} = \bar{x} \pm tS_{\bar{x}}$$

	$\frac{S_x^2}{N}$	N	DF	t	$\frac{S_x^2}{N}$	\bar{x}	$tS_{\bar{x}}$
Formulation	6.45	2	6	2.45	2.63	1.62	3.96
Priming	6.45	7	6	2.45	.923	.963	2.35

ANALYSIS OF VARIANCE

SOURCE	DF	SS	MS	F
TOTAL	13	8.08571428580e+01		
INTER	6	3.87142857164e+01	6.45238095270e+00	
I	6	2.39571428582e+01	3.97619047637e+00	
INTER	6	3.87142857164e+01	6.45238095270e+00	6.16236162360e-01
J	1	1.82857142836e+01	1.82857142836e+01	
INTER	6	3.87142857164e+01	6.45238095270e+00	2.83394833900e+00

NO FACTORS SIGNIFICANT

I	J	MEAN
0	0	7.71428571430E+00
0	1	6.57142857140E+00
0	2	8.85714285720E+00
1	0	6.00000000000E+00
1	1	2.00000000000E+00
1	2	1.00000000000E+01
2	0	5.50000000000E+00
2	1	2.00000000000E+00
2	2	9.00000000000E+00
3	0	8.00000000000E+00
3	1	8.00000000000E+00
3	2	8.00000000000E+00
4	0	8.50000000000E+00
4	1	8.00000000000E+00
4	2	9.00000000000E+00
5	0	8.00000000000E+00
5	1	8.00000000000E+00
5	2	8.00000000000E+00
6	0	9.00000000000E+00
6	1	9.00000000000E+00
6	2	9.00000000000E+00
7	0	9.00000000000E+00
7	1	9.00000000000E+00
7	2	9.00000000000E+00

ANALYSIS OF VARIANCE

SERIES II

Night Visibility Only
Concrete Only
Exposure 12 Months

<u>Letter</u>	<u>Independent Variable</u>	<u>Variable Identification</u>	
I	Formulation	1 Polyamide #155	5 Alkyd #162
		2 Polyamide #157	6 Alkyd #163
		3 Polyamide #158	7 Proprietary #164
		4 1 S/A #160	
J	Priming	1 None	
		2 Epoxy	

Computation of Confidence Limits

$$S_{\bar{x}} = \sqrt{\frac{S^2}{N}} ; 95\% \text{ Confidence Range} = \bar{x} \pm tS_{\bar{x}}$$

	$\frac{S^2}{x}$	$\frac{N}{x}$	$\frac{DF}{x}$	$\frac{t}{x}$	$\frac{S^2}{x}$	$\frac{S}{x}$	$\frac{tS}{x}$
Formulation	.833	2	6	2.45	.415	.645	1.58
Priming	.833	7	6	2.45	.119	.346	.846

ANALYSIS OF VARIANCE

SOURCE	DF	SS	MS	F
TOTAL	13	1.235000000000E+02		
INTER	6	5.000000000000E+00	8.33333333340E-01	
I	6	8.700000000000E+01	1.450000000000E+01	
INTER	6	5.000000000000E+00	8.33333333340E-01	1.740000000001E+01 ***
J	1	3.149999999999E+01	3.149999999999E+01	
INTER	6	5.000000000000E+00	8.33333333340E-01	3.780000000000E+01 ***

***SIGNIFICANT AT 1% LEVEL

I	J	MEAN
0	0	9.500000000000e+00
0	1	1.100000000001e+01
0	2	8.000000000000e+00
1	0	8.500000000000e+00
1	1	1.000000000000e+01
1	2	7.000000000000e+00
2	0	1.350000000001e+01
2	1	1.399999999999e+01
2	2	1.300000000000e+01
3	0	9.000000000000e+00
3	1	1.000000000000e+01
3	2	8.000000000000e+00
4	0	8.500000000000e+00
4	1	1.100000000001e+01
4	2	6.000000000000e+00
5	0	1.149999999999e+01
5	1	1.300000000000e+01
5	2	1.000000000000e+01
6	0	1.050000000000e+01
6	1	1.200000000000e+01
6	2	9.000000000000e+00
7	0	5.000000000000e+00
7	1	7.000000000000e+00
7	2	3.000000000000e+00

B. Test Methods and Specifications

Research Test Methods, Routine and Special

Recommended Specifications

Specifications From Other Sources

CONSISTENCY DETERMINATION

Apparatus:

1. Brookfield Viscosimeter RTV with extension rod.
2. Heavy-duty hot plate.
3. Metal Thermometer (100-500° F).
4. No. 303 Sanitary Cans.

Procedure:

1. Heat specimen slowly with stirring to 460° F.
2. Introduce Spindle #6 into melt, operate at 20 RPM.
3. Remove hot plate, observe and record temperature and viscosities as the melt cools slowly to 300° F.
4. Plot data on log viscosity versus reciprocal absolute temperature coordinates.
5. Read off consistency and slope (consistency change per °F) at 350° F.

ELASTIC MODULUS IN FLEXURE, ELASTIC DEFORMATION, AND CREEP

Apparatus

The Creep Tester is shown in Figure 23 page 163. Testing is performed on specimens 0.5" x 0.5" in cross section and at least 5" long. The test span is 4". The standard loading is 3356 g.

Modulus

An approximate flexural elastic modulus was determined on the creep test device. The procedure involved loading a specimen with a fixed weight for about five seconds, observing the deflection, unloading, and immediately observing the recovery deflection. The difference in deflections is taken as the elastic deformation and

$$E_f = \frac{L^3}{4bd^3} \frac{P}{\delta}$$

where: E_f = modulus of elasticity in flexure, psi

L = span, in.

b = beam width, in.

d = beam depth, in.

P = load, lbs.

δ = deflection difference, in.

Creep Rate

The creep characteristics of various formulations have been determined in the form of creep curves (deflection versus time at a fixed load). Similarly to consistency, these data have been condensed into two parameters for

general characterization. The primary parameter is the Creep Rate which is taken arbitrarily as the slope of the deflection versus time curve at one minute. The Creep Rate is presented in units of

$$\frac{\text{in. (deflection)}}{\text{in. (gage length) min}} = \text{min}^{-1}$$

Elastic Deformation

The secondary parameter is the instantaneous elastic deformation which represents the zero time intercept of the creep curve with the deflection ordinate. Its units are

$$\frac{\text{in. (deflection)}}{\text{in. (gage length)}}$$

One notes that the creep rate and elastic deformation are functions of the constant maximum fiber stress applied, whereas the elastic modulus is a property that is independent of the loading.

IMPACT TESTS

Apparatus

A flywheel impact tester was modified to accomodate the very low impact energy levels that characterize the hot melts. This device is shown in Figure 15. With this lightweight flywheel, the total energy of the device was only seven inch-pounds.

Procedure

The determinations are made from unnotched Charpy-type specimens $\frac{1}{2}$ " x $\frac{1}{2}$ " in cross-section on a span of 3.25 inches. Maximum fiber stress was computed from the impact load data in the same manner as for a static flexural strength test, thus

$$s = \frac{3PL}{2bd^2}$$

where: s = maximum fiber stress, lbs/in²

P = load, lbs.

L = span, in.

b = beam width, in.

d = beam depth, in.

Observed impact energy was converted to a basis of one square inch of specimen cross-section so the units of the test become in-lbs/in².

$$E = \frac{7R(\%)}{.25 \text{ in}^2} \text{ in-lbs}$$

where: E = Impact Energy, in-lbs

R = Scale Reading

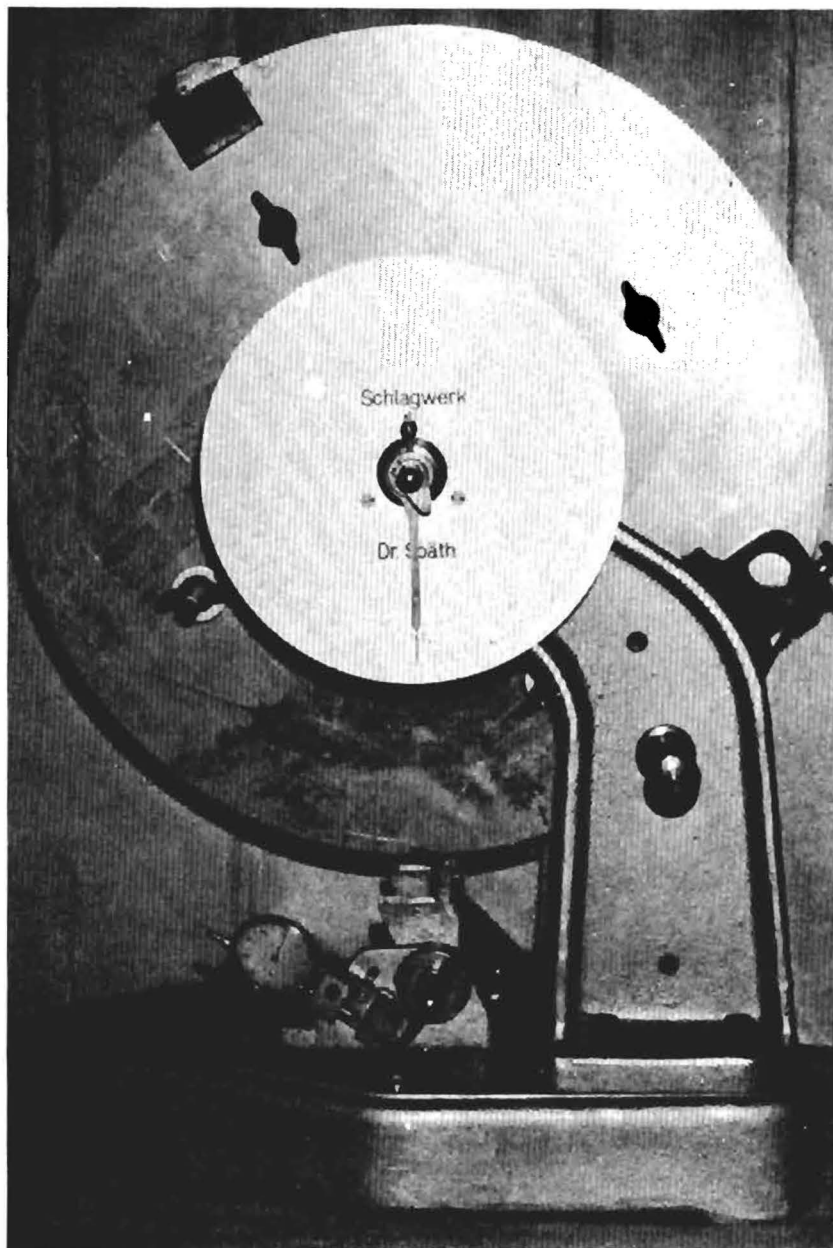


Figure 15. Impact tester.

SHEAR BOND STRENGTH TEST

Apparatus

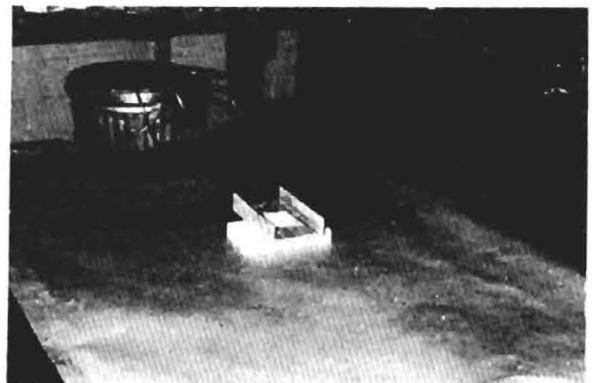
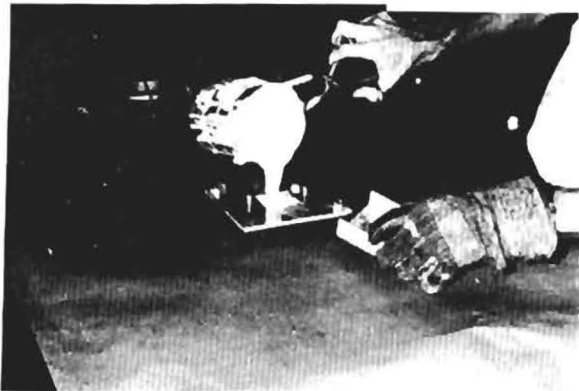
Special shear fixture made to be used with a 20 ton Carver Hydraulic Press.

Procedure

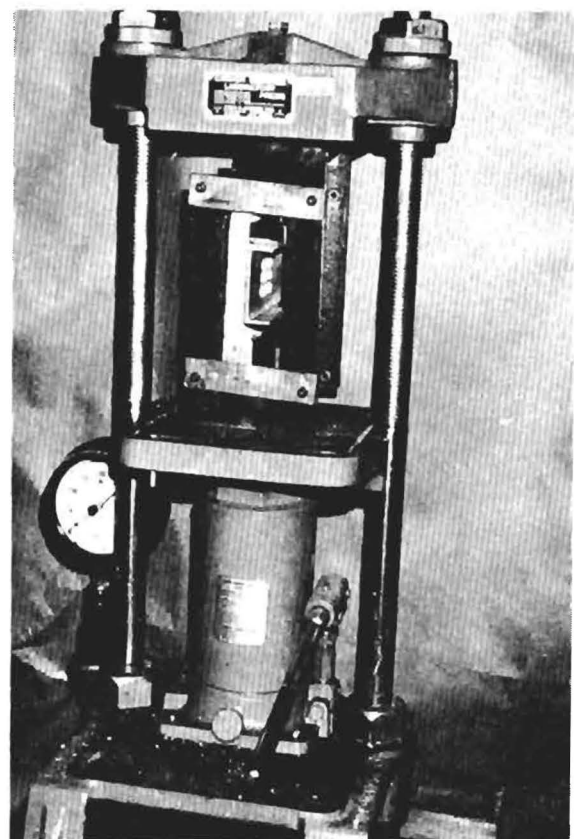
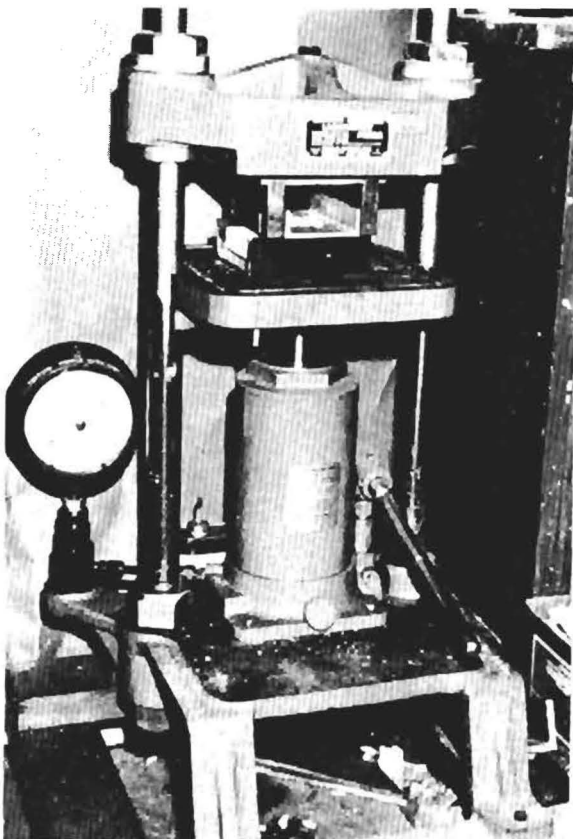
The hot melt is molded to dimensions of 2 x 2 inches in cross-sections and 0.25 inches thick between a concrete block and a perforated aluminum channel. The use of the perforated channel insures a disbond only at the concrete interface. The bonded sample is placed in the shear device and shear force applied. This maximum shear force is recorded and the force in pounds needed to disrupt the bond is divided by 4 square inches of area to yield the pounds per square inches strength.

$$\text{psi} = \frac{F(\text{pounds})}{4(\text{sq.in.})}$$

where: F = force, lbs



MOLDING SPECIMENS



BOND TESTING

Figure 16. Bond strength - preparation and testing.

FLOWABILITY TEST

Procedure

A sample of 400 gm in a No. 303 sanitary can is heated to 450° F with stirring. After stirring rapidly for 10 seconds the can is emptied at a 45° angle. Samples not rapidly stirred were also run.

Results

<u>Sample</u>	<u>% Retained Stirred</u>	<u>% Retained Not Stirred</u>
Polyamide Low #158	>1	5.0
Polyamide Med. #155	>1	5.5
Polyamide High #157	>1	3.5
Polyamide High (Heat Treated) #157-T	17.2	9.2
Cal. Spec. #168	>1	9.2

Comments

This test was examined because of its use in specification by other authorities and with the thought that it might characterize a practical low-shear consistency. The results were very sensitive to stirring effects and did not appear to yield the characterizing information desired.

THERMAL DECOMPOSITION OF HOT MELTS

Various Hot Melts were compared for thermal stability by placing 20 gm of each in an aluminum weighing dish and heating to approximately 750° F on a surface heated hot plate. After 5 minutes the following conditions were observed.

($\frac{1}{4}$ TiO₂) HM #42 (Reg. Alkyd) - badly yellowed - vehicle decomposing
($\frac{1}{4}$ TiO₂) HM #65 (Ethocel w/Eastobond) - very brown - vehicle decomposing
($\frac{1}{4}$ TiO₂) HM #56 (Ethocel) - very brown - vehicle decomposing
($\frac{1}{4}$ TiO₂) HM #57 (Stabilite) - beginning to yellow - vehicle still effective
(Full TiO₂) HM #56 (Cal. Spec.) - still white - no apparent damage to vehicle

Comments

This test appeared to provide a rapid indication of thermal stability-- potentially useful for quick comparisons of materials.

CREEP BEHAVIOR

It was of interest to determine if our hot melts obey the Nutting Equation for creep:

$$E = K\sigma t^n$$

or

$$\log E = \log K + \log \sigma + n \log t$$

where: E = elongation

K = a constant (degree of flexibility)

σ = stress

t = time

n = a constant (degree of viscous response)

If this equation is obeyed, a plot of creep versus time on log-log scales will yield a straight line of slope n . Since stress is constant under test conditions, at any time, t , the value of K may be determined from the displacement (elongation). This provides a two-parameter (n , K) characterization of creep behavior.

Plots of several formulations are shown in Figure 17. The curves are straight lines and do display reasonable systematic trends of flexibility. Viscous response is seen to be fairly constant among the formulations excepting #157. In this case the distinctly lower viscous response may be attributable to the high polyamide content.

It was concluded that the Nutting Equation is applicable to our materials, and the log-log plots are helpful for characterizing and comparing creep behavior.

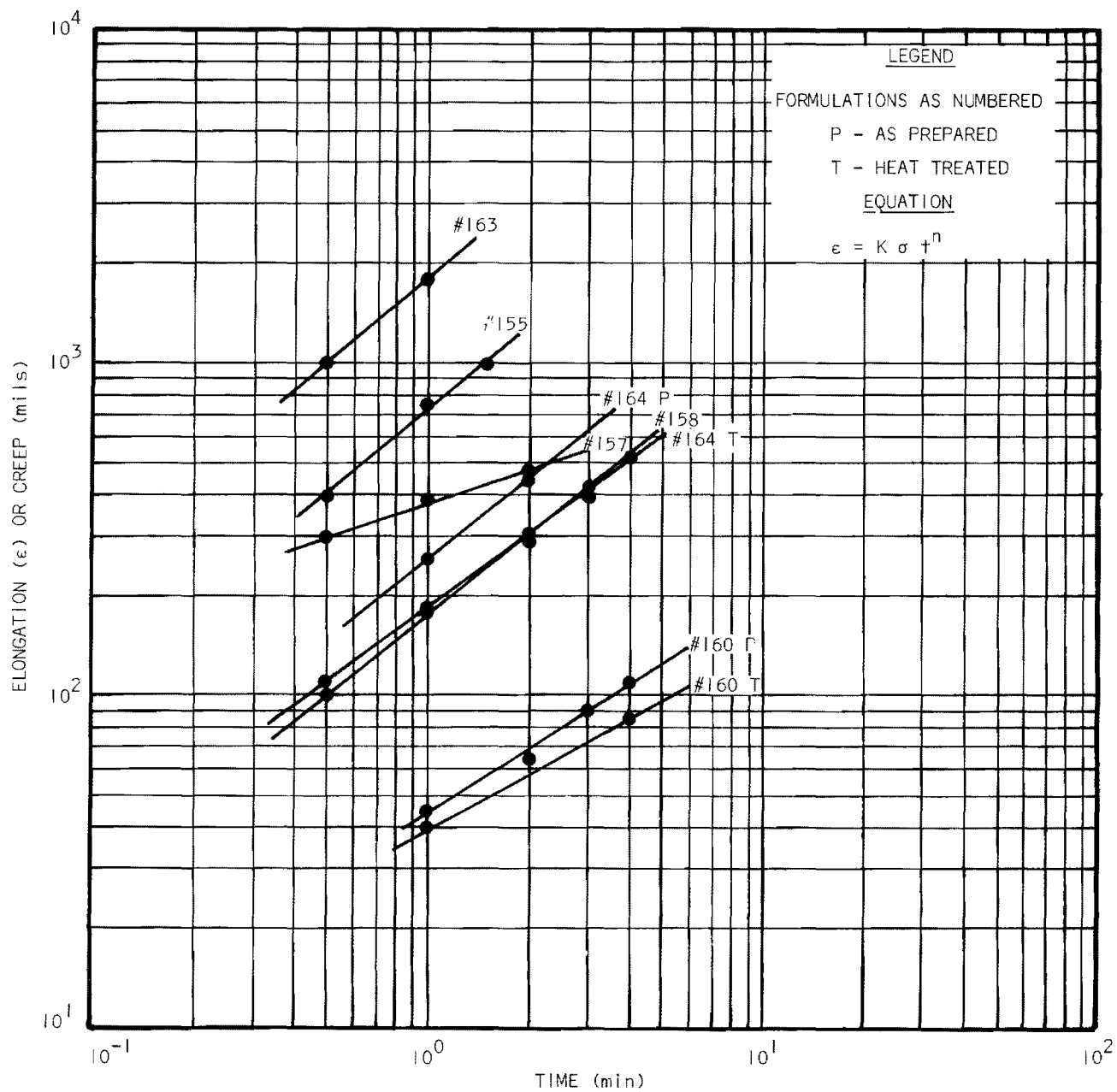


Figure 17. Plot of nutting equation.

STATE HIGHWAY DEPARTMENT OF GEORGIA
TENTATIVE MAINTENANCE SPECIFICATION
FOR
CLEAR PRIMER FOR HOT MELT TRAFFIC LINES

General

This tentative specification was developed under Research Project No. A-802 (Contract No. 6401) based upon experimental findings and currently available technical information. It is requested that recommendations for changes be directed to the State Highway Materials Engineer of the State Highway Department of Georgia.

No prior specifications have been issued covering the product and application described hereunder.

Scope and Classification

Scope - This primer is intended for use on Portland cement and asphaltic concrete surfaces prior to the application of thermoplastic hot melt traffic lines.

Classification - Clear primer shall be of the following types as specified in the invitation for bids, contract or order.

Type I - Synthetic rubber - resin type

Type II - Epoxy - polyamide type, two package,
Parts A and B

Requirements

Materials

Materials shall be as herein specified. Any materials not definitely specified shall be of good commercial quality.

Primer Composition

The primer components shall be as specified in Table I and below for the respective types of primer.

Type I - This primer shall be supplied in a single package, and shall consist of an intimate blend of Buna N rubber with a compatible phenolic resin in a solution of the specified solvents.

When dried upon a potassium bromide disk, a film of the primer shall have infrared absorption maximums at the same wavelengths and to the same relative degree as that shown by the curve of Figure 18.

TABLE I
PRIMER COMPOSITION
(Percent by weight of primer)

<u>Ingredients and Specifications</u>	<u>Type I</u>	<u>Type II</u>	
		<u>Part A</u>	<u>Part B</u>
Buna N rubber	6-8		
Phenolic resin	3-4		
Epoxy liquid resin		50 min.	
Polyamide resin			50 min.
Methyl ethyl ketone (TT-M-261)	45 max.		
Methyl isobutyl ketone (TT-M-268)	18 max.		
Diisobutyl ketone	4.5 max.		
Toluene (TT-T-548a)	18 max.		
Xylene (TT-X-916)	4.5 max.		
Acetone (ASTM D329-58)		50 max.	50 max.

Type II - This primer shall be supplied in two packages of approximately equal volume. Part A shall consist of a liquid epoxy resin, viscosity 150 poises max. (at 25° C), epoxide equivalent 175-210; a bisphenol-type resin exhibiting an infrared spectrum as shown in Figure 19. It shall be supplied in an acetone solution. Part B shall consist of a liquid polyamide resin, viscosity 7-9 poises (at 75° C), amine value 290-320 (mg. KOH/g sample); a dimerized fatty acid - aliphatic polyamide-type resin exhibiting an infrared spectrum as shown in Figure 20. It shall be supplied in an acetone solution.

Primer Qualitative Requirements

Condition in Container - In a freshly-opened full can the primer shall appear smooth and homogeneous with no skins or gel particles.

Storage Stability - When stored up to six months from date of delivery, the primer shall exhibit no appearance change as compared with initial condition in container.

Mixing Properties, Type II - When Parts A and B are mixed in equal volumes a smooth homogeneous mixture shall result. When the mixture is aged in a filled, closed container for eight hours it shall show no signs of gelation.

Spraying Properties - The primer shall spray satisfactorily in all respects, and show no signs of cobwebbing or dusting.

Sampling, Inspection, and Testing

Unless otherwise specified, sampling, inspection and testing shall be in accordance with Method 1031 of Federal Standard 141. Samples shall be

submitted with each bid. Each sample shall consist of one-half gallon of primer submitted in quart cans (one quart each of Parts A and B, Type II).

Packaging

Clear primer for hot melt traffic lines shall be delivered in 55 gallon drums unless otherwise specified. The description, requisition number, purchase order number, gross and net weights shall be stenciled on the head of each drum. The drums shall be of the removable head type.

Certified Analysis

The manufacturer shall submit with his bid sample a certified analysis setting forth the composition of the primer. It shall include the following information: Percentage composition of the primer to include brand and kind of ingredients. It shall contain the actual test data obtained by the manufacturer in determining compliance with the physical tests required by these Specifications. In addition, the manufacturer shall further certify that if awarded the contract the material furnished will be identical with that of the sample submitted. This information shall be held as confidential.

Patents

The Contractor shall assume all costs arising from the use of patented materials, equipment, devices, or processes used on or incorporated in the work, and agrees to indemnify and save harmless the State of Georgia, and their duly authorized representative from all suits at law or action of every nature for, or on account of the use of any patented materials, equipment, devices or processes.

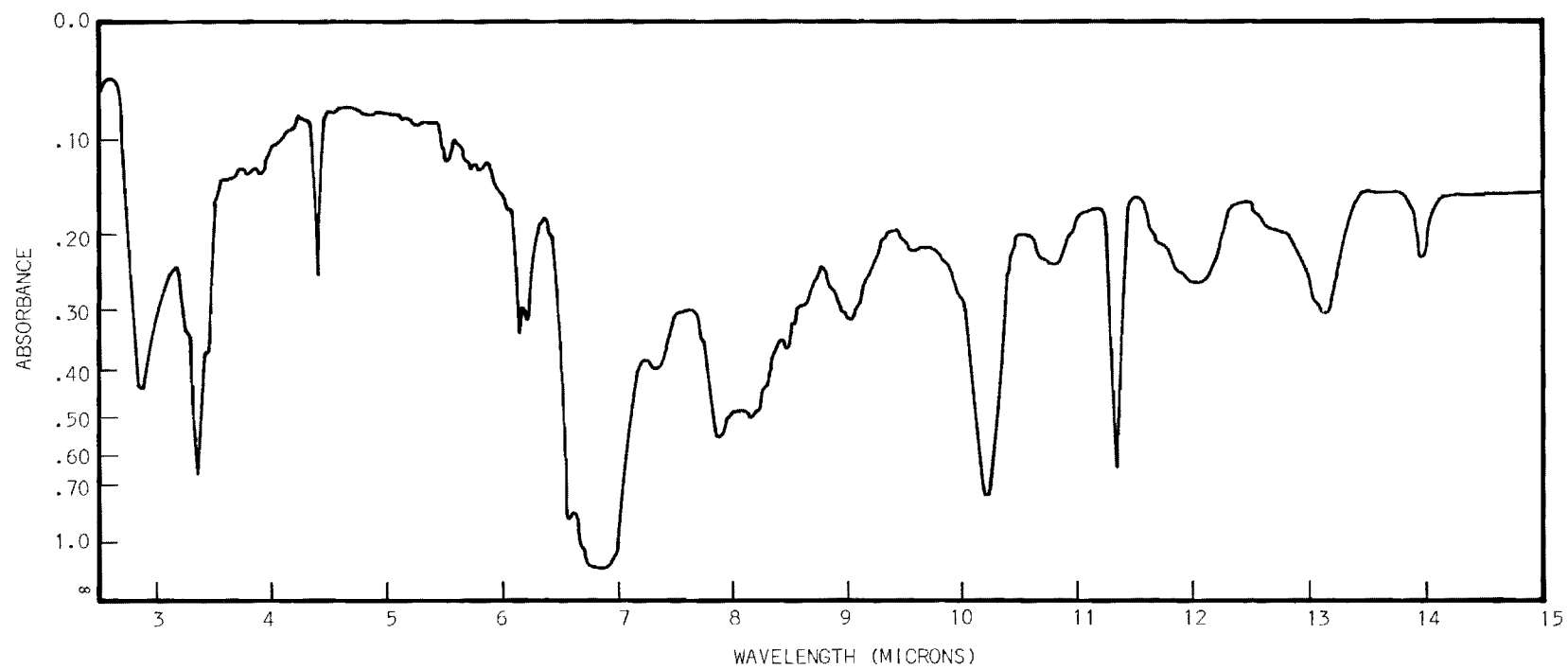


Figure 18. Buna N - phenolic resin infrared spectrum.

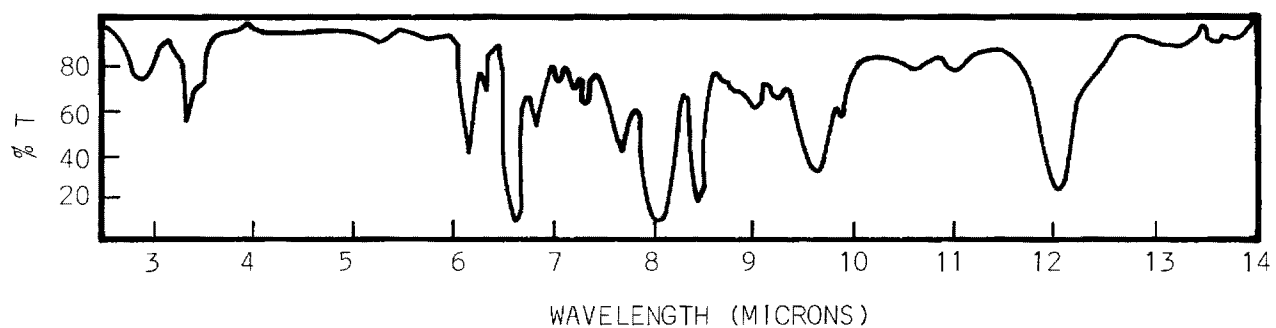


Figure 19. Bisphenol type epoxy resin infrared spectrum.

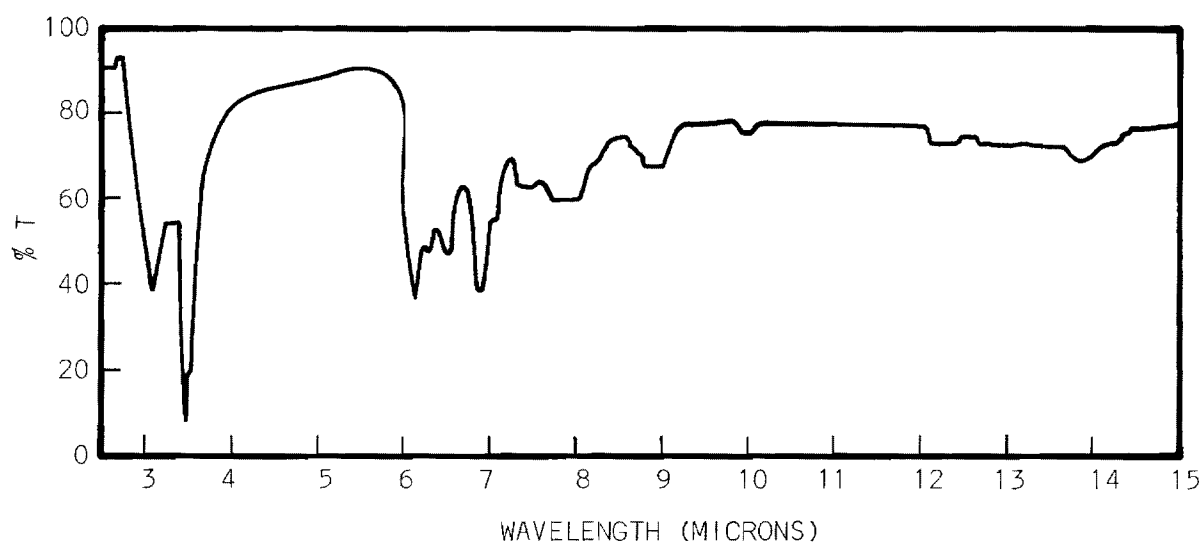


Figure 20. Dimerized fatty acid - aliphatic polyamine-type resin infrared spectrum.

STATE HIGHWAY DEPARTMENT OF GEORGIA

TENTATIVE SPECIFICATION

FOR

THERMOPLASTIC TRAFFIC STRIPE

General

This tentative specification was developed under Research Project No. A-802 (Contract No. 6401) based upon experimental findings and currently available technical information. It is requested that recommendations for changes be directed to the State Highway Materials Engineer of the State Highway Department of Georgia.

No prior specifications have been issued covering the product and application described hereunder.

Scope and Classification

Scope - This is a performance specification for thermoplastic traffic striping material which is applied while molten with immediate drop on application of glass beads. Upon cooling to pavement temperature the material solidifies to form a durable retroreflective stripe.

Classification - The thermoplastic material shall be of the following two types as specified in the invitation for bids, contract, or order.

Type I - White

Type II - Yellow

Requirements

Materials - The manufacturer is granted wide latitude in selecting materials and in formulating to meet the performance requirements of this specification.

Conditioning Requirements - All specimens of hot melt material selected for testing shall first be heat aged as specified below. In addition, a water freeze cycle treatment is specified for use with tensile bond strength and impact tests.

	<u>Heat Aging Treatment</u>
Temperature	- 400 \pm 10° F
Time	- 4 hours
Agitation	- Slow, continuous, approx. 250 RPM

The material for treatment is melted in a heated, 1 quart stainless steel beaker with a propeller agitator that is set close to the bottom. The beaker should be at least half filled with molten material. Timing begins when the specified temperature is reached. Test specimens should be prepared immediately following conditioning.

Water-Freeze Cycle Treatment

Specimens are placed in a water bath maintained at 77° F for 18 hours. The specimens are then transferred to a freezer maintained at 0° F for 3 hours. The specimens to be tested at 0° F are tested as soon as they are removed from the freezer. The specimens to be tested at 77° F are returned to the water bath for 2 hours and then tested.

Performance Requirements - The performance requirements of both Type I and Type II materials shall be identical unless otherwise specified.

Consistency

Consistency of hot melts is measured using a Brookfield viscometer, Model RTV, 20 RPM, and an oil bath maintained at 350° F. The sample of

approximately 800 g is placed in a Size 303 can, heated to a uniform temperature slightly above 350° F and placed in the oil bath. A Brookfield spindle is warmed and then placed in the sample and moved around to bring it to the temperature of the hot melt and release any air trapped under it before attaching it to the viscometer. The reading is taken when the temperature of the hot melt has reached 350° F. The sample should be carefully stirred just prior to taking the reading to insure uniformity. The consistency shall be no more than 35,000 cps, nor less than 2,000 cps.

Flexural Creep Test

The flexural creep of a hot melt is obtained by measuring the sagging displacement of a 1/2 x 1/2 x 6 in. bar of hot melt which is suspended over supports 4 inches apart, and a load of 3,356 grams applied to the center of the bar. The sagging is measured by a dial micrometer, as shown in Figure 23.

The mils of sagging are plotted against time on linear graph paper and the slope at one minute is taken as the flexural creep. This slope shall not exceed 30×10^{-3} in./in./min.

Static Indentation Test

The static indentation is obtained by measuring the diameter of the impression left by a 3/4 in. diameter steel ball loaded to 200 grams for 24 hours on a hot melt disk prepared as described in the Falling Ball Impact Test. The measured diameter of indent should not exceed 200 mils or 0.20 of an inch. A test assembly is shown in Figure 21.

Tensile Bond Strength

Specimens for the tensile bond strength of a hot melt are obtained by cementing two 3 x 5 x 3/4 in. concrete blocks together with a 2 x 2 x 1/4 in.

layer of hot melt at 350° F in such a manner that the long dimensions of the block are at 90° to each other. Before the cementing operation, the faces of the concrete blocks are sandblasted to remove laitance. The samples are subjected to the water-freeze cycles as a part of the testing procedure. Specimens are tested in a hydraulic press for tensile bond strength. (The sample should be placed in the press in such a manner that the only forces on the bond are tensile forces.) The tensile bond strengths shall be an average of 5 tested specimens at each temperature and the averages shall equal or exceed 15 psi at 77° F and 50 psi at 0° F.

Falling Ball Impact Test

Prepare 10 disks from heat-aged hot melt which are 0.25 ± 0.025 in. thick and approximately 2 in. in diameter.* These disks should not vary more than $\pm 5\%$ in weight. The disks are subjected to the water-freeze cycle as a part of the testing procedure.

A disk is placed on an anvil which is a 3 pound block of brass 2 in. in diameter with the top covered by a $1/16$ in. layer of rubber.

A $1/2$ in. diameter steel ball is dropped onto the disk from a position one foot above and centered on the disk. Failure is considered to have occurred when a crack appears in the sample. Four of the five disks must withstand three drops of the steel ball without failure for the material to pass. A test assembly is shown in Figure 22.

* Aluminum Foil Cups, Fisher No. 8-732, are satisfactory for molds.

Color, Type II, Yellow

The color of Type II, after cooling to 77° F, shall match Color No. 1315 of Federal Specification TT-C-595.

Notes on Application

The thermoplastic traffic striping is intended to be used with a specified primer on dry, clean concrete surfaces. On application to asphalt surfaces, no primer is required. The application should be made at environmental temperatures no lower than 65° F.

Sampling and Testing

Samples shall accompany each bid. Each sample shall consist of a 50 lb. standard package of material. The right is reserved to inspect and sample all lots submitted for use and to inspect the manufacturing process.

Packaging

The thermoplastic traffic striping material shall be packaged in suitable containers to which it will not adhere during shipment or storage. The blocks of cast thermoplastic material shall be approximately 12 x 35-1/2 x 2 inches and shall weigh approximately 50 pounds. Each container label shall designate the color, manufacturer's name, batch number, and date of manufacture.

Certified Data Report

The manufacturer shall submit with his bid a certified test data report. It shall contain the actual test data obtained by the manufacturer in determining compliance with the physical tests required by these specifications. In addition, the manufacturer shall further certify that if awarded the

contract the material furnished will be identical with that of the sample submitted. This information shall be held as confidential.

Patents

The Contractor shall assume all costs arising from the use of patented materials, equipment, devices, or processes used on or incorporated in the work, and agrees to indemnify and save harmless the State of Georgia, and their duly authorized representative from all suits at law or action of every nature for, or on account of the use of any patented materials, equipment, devices or processes.

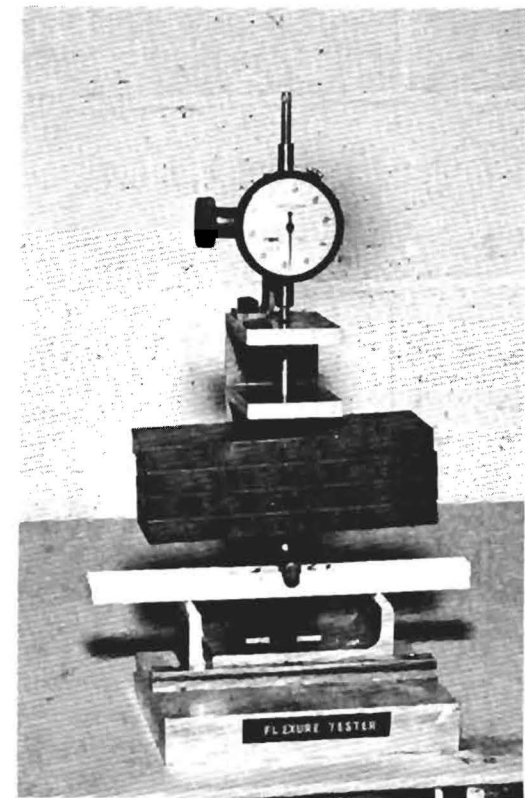
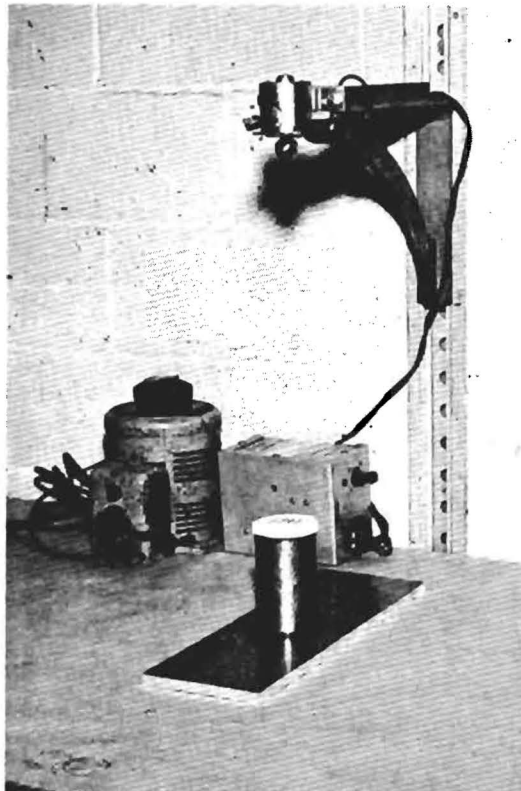


Figure 21. Static indentation test. Figure 22. Falling ball impact test.

Figure 23. Flexure test.

State of California
Department of Public Works
Division of Highways

SPECIFICATION

1963

PRIMER FOR TRAFFIC PAINTS
AND THERMOPLASTIC TRAFFIC PAINTS

Scope

This primer is intended for use on new Portland cement concrete highway surfaces prior to application of traffic paint and on Portland cement and asphaltic concrete surfaces prior to the application of thermoplastic traffic paint.

Composition

Buna N rubber and phenolic resin

Buna N Rubber/Phenolic Resin Ratio
Between 60/40 and 70/30

Percent Solids

9 to 11%

Composition of Solvent Used

50% methyl ethyl ketone

20% methyl isobutyl ketone

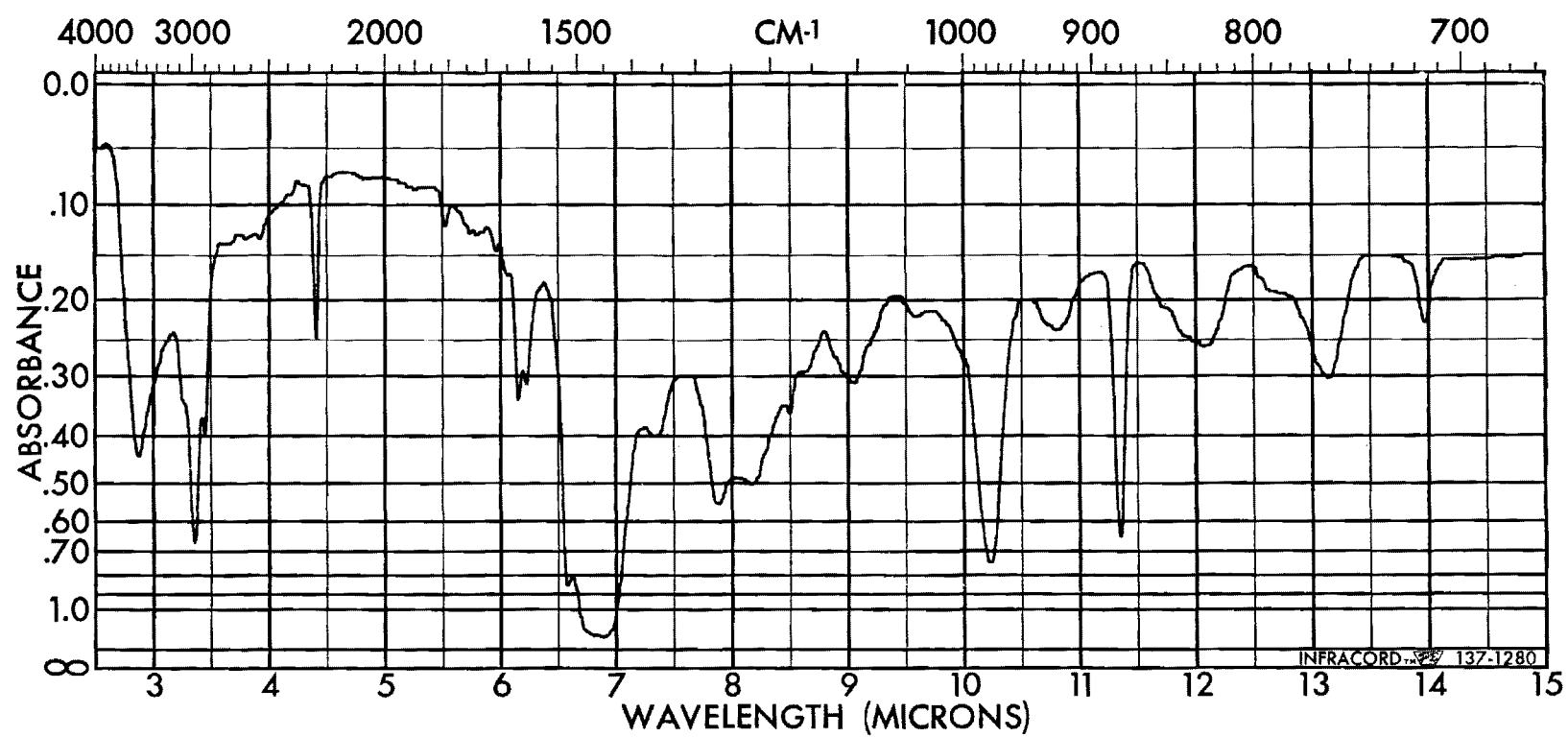
20% toluene

5% diisobutyl ketone

5% xylene

Infrared Characteristic Curve of Primer

When dried upon a potassium bromide disc, a film of the primer shall have infrared absorption maximums at the same wavelengths and to the same relative degree as that shown by the following curve.



State of California
Department of Public Works
Division of Highways

Materials and Research Department

SPECIFICATION

1964

EXPERIMENTAL WHITE THERMOPLASTIC TRAFFIC LINE
FORMULA 1R353

Description

These specifications cover a reflectorized thermoplastic pavement striping material of a type that is applied to the road surface in a molten state by mechanical means with surface application of glass beads and, which upon cooling to normal pavement temperature, produces an adherent reflectorized stripe of specified thickness and width and capable of resisting deformation by traffic.

White Type

Composition

The thermoplastic pavement marking material shall contain the following ingredients in the designated percentage composition by weight.

	<u>Per Cent by Wt.</u>
Reichhold hydrogenated ester gum, "Hydrogum P-190" or equal	9.8
Goodyear Pliolite AC1 Milled or equal	9.0
Hercules hydroabietyl alcohol, "Abitol" or equal	5.8
Glass Beads	20.8
Titanium Dioxide, Federal Specification TT-P-442, Type III, Class A	13.0
C. K. Williams Marble White, 50 mesh or equal ¹	41.6

The resins should be heated to about 464° F in such a manner as to prevent localized overheating and under rapid agitation to insure complete mutual solubility prior to adding the other ingredients. Any manufacturer not familiar with the technique of making this material should consult the Materials and Research Department of the Division of Highways. A small trial batch should be made prior to making the thermoplastic traffic line in large quantities to make certain the finished product will comply with all the requirements of this specification.

The pigment, beads and filler shall be well dispersed in the resin. The material shall be free from all skins, dirt and foreign objects.

The glass beads used in the formulation shall have a refractive index of not less than 1.50 when tested by the liquid immersion method at 25° C; shall consist of 75% minimum by count of water white true spheres; shall be free from air inclusions and shall have the following grading:

<u>Sieve Number</u>	<u>Percent Passing</u>
40	95 minimum
70	10 maximum

Physical Characteristics of the Thermoplastic Material

The material shall not exude fumes which are toxic, obnoxious or injurious to persons or property when it is heated to 450° F. It shall remain stable when held for four hours at this temperature, or when reheated repeatedly after cooling to the ambient temperature.

The temperature-viscosity characteristics of the plastic material shall remain constant throughout repeated reheatings, and shall show

like characteristics from batch to batch. There shall be no obvious change in color of the material as a result of repeated reheatings or from batch to batch.

When applied at a temperature range of 400-450° F and thickness of 1/8 inch to 3/16 inch, the material shall set to bear traffic in not more than two minutes when the air temperature is 50° F and not more than ten minutes when the air temperature is 90° F.

The thermoplastic material shall readily extrude at 400° F from the equipment used by the Division of Highways to produce a cross-section of line 1/8 inch to 3/16 inch thick which shall be continuous and uniform in shape, and have clear and sharp dimensions.

After heating for four hours at 450° F, the thermoplastic traffic line material shall conform to the following requirements:

Reflectance:

The daylight luminous reflectance of the material shall not be less than 75% relative to magnesium oxide, after cooling to 77° F, when tested according to ASTM E 97.

Bond Strength:

When two concrete blocks, 2 x 3-1/2 x 7 inches, the blocks being at 77° F, are cemented together on the 3-1/2 inches by 7 inches faces with a 1/16 inch to 1/8 inch thick layer of the thermoplastic traffic line material and tested according to ASTM C 321, the bond strength at 77° F shall not be less than 200 pounds per square inch. Failure shall be a minimum of 50% in cohesion of the material as measured on the total

area of the break. The faces of the concrete blocks to which the thermoplastic traffic line is applied shall be first sandblasted to remove laitance and primed with the Primer for Traffic Paint and Thermoplastic Traffic Paints, State Specification 63-F-40.

Yellowness Index:

When tested according to Federal Test Method Standard No. 141, Method 6131, the yellowness shall not exceed 0.12 after the sample is cooled to 77° F.

Indentation Resistance:

Hardness shall be measured by a Short Durometer, Type A2, as described in ASTM D 1706-59T, except that the Durometer shall be at 75-77° F, and a 2 kilogram load applied (Wt. of Durometer and load 2170 ± 30 gms.). After 15 seconds, the reading shall be the amount herein designated when the material is cooled to the following temperature:

<u>Temperature</u>	<u>Reading</u>
115° F	47-57

Flowability:

When 400 grams of the thermoplastic traffic line is poured at a 45° angle from a cylindrical pint can approximately 3-3/4 inches high and 3-5/16 inches in diameter and with the rim removed, the residue in the can after flow ceases shall be 13 to 17 percent of the original weight when the melted material is stirred rapidly for 10 seconds before pouring.

Low Temperature Stress
Resistance (Cracking):

The thermoplastic material shall not crack or fail to adhere to the substrate when tested as follows:

A 1/8 inch thick layer of the thermoplastic material shall be applied to a one-foot square surface of a Portland cement concrete block 4 inches thick which surface has been first sandblasted to remove all laitance and foreign matter and primed with the Primer for Traffic Paints, State Specification 63-F-40. After the specimen has reached $77 \pm 5^{\circ}$ F, it shall be placed in a freezer chest or other insulated cold compartment and maintained at 15° F for 24 hours. The thermoplastic material shall be examined continuously for 5 minutes immediately after the concrete block is removed from the cold compartment.

After heating for four hours at 400° F, the thermoplastic traffic line material shall conform to the following requirements:

Flowability:

A maximum of not more than 20% residue shall remain in the can when tested according to the procedure outlined above for Flowability at 450° F.

Packaging:

The thermoplastic traffic line shall be packaged in suitable containers to which it will not adhere during

shipment or storage. The blocks of cast thermoplastic material shall be approximately 12 x 35-1/2 x 2 inches and shall weigh approximately 60 pounds. Each container label shall designate the color, manufacturer's name, batch number, date of manufacture and the notation "Experimental White Thermoplastic Traffic Line, Formula 1R354." Each Batch manufactured shall have its own separate number.

Inspection:

Each batch of the thermoplastic traffic line paint manufactured shall be subject to inspection and approval by the Materials and Research Department of the Division of Highways before acceptance. The State reserves the right to have an inspector present to observe the manufacturing process. The vendor shall furnish a complete formulation record of his manufacturing process to the Materials and Research Department.

Patents:

The Contractor shall assume all costs arising from the use of patented materials, equipment, devices, or processes used on or incorporated in the work, and agrees to indemnify and save harmless the State of California, and their duly authorized representative from all suits at law or action of every nature for, or on account of the use of any patented materials, equipment, devices or processes.

State of California
Department of Public Works
Division of Highways

Materials and Research Department

SPECIFICATION

1964

THERMOPLASTIC TRAFFIC LINE

Description

These specifications cover a reflectorized thermoplastic pavement striping material of a type that is applied to the road surface in a molten state by mechanical means with surface application of glass beads and which upon cooling to normal pavement temperature, produces an adherent reflectorized stripe of specified thickness and width and capable of resisting deformation.

White Type

Composition

The thermoplastic pavement marking material shall contain not less than 20 percent by weight of glass spheres and not less than 12 percent by weight of titanium dioxide.

The pigment, beads and filler shall be well dispersed in the resin. The material shall be free from all skins, dirt and foreign objects. It shall be of such composition that it will not bleed, stain or discolor when applied to bituminous pavements.

The glass beads used in the formulation shall have a refractive index of not less than 1.50 when tested by the liquid immersion method at 25° C;

shall consist of 75% minimum by count of water white true spheres; shall be free from air inclusions and shall have the following grading:

<u>Sieve Number</u>	<u>Percent Passing</u>
40	95 minimum
70	10 maximum

Physical Characteristics of
the Thermoplastic Material

The material shall not exude fumes which are toxic, obnoxious or injurious to persons or property when it is heated to the temperature range specified by the manufacturer for application. It shall remain stable when held for four hours at this temperature, or when reheated repeatedly after cooling to the ambient temperature.

The temperature-viscosity characteristics of the plastic material shall remain constant throughout repeated reheatings, and shall show like characteristics from batch to batch. There shall be no obvious change in color of the material as a result of repeated reheatings or from batch to batch.

When applied at the specified temperature and thickness, the material shall set to bear traffic in not more than two minutes when the air temperature is 50° F and not more than ten minutes when the air temperature is 90° F.

The thermoplastic material shall readily extrude at 400° F from the equipment used by the Division of Highways to produce a cross-section of line 1/16 inch to 1/8 inch thick which shall be continuous and uniform in shape, and have clear and sharp dimensions.

After heating for four hours at 450° F, the thermoplastic traffic line material shall conform to the following requirements:

Reflectance

The daylight luminous reflectance of the material shall not be less than 75% relative to magnesium oxide after cooling to 77° F when tested according to ASTM E 97.

Impact Resistance

The impact resistance shall not be less than 10 inch pounds at 77° F after the material has been cast into bars of 1 inch cross-sectional area and 3 inches long and placed with 1-1/4 inch extending above the vise in a cantilever beam (Izod type) tester using the 25 inch pound scale. See ASTM D 256 for description of this instrument, except that the striking edge of the pendulum shall be 1.058 inch above the top surface of the vise.

Bond Strength

When two concrete blocks, 2 x 3-1/2 x 7 inches, the blocks being at 77° F, are cemented together on the 3-1/2 inches by 7 inches faces with a 1/16 inch to 1/8 inch layer of the thermoplastic traffic line material and tested according to ASTM C 321, the bond strength at 77° F shall not be less than 150 pounds per square inch. Failure shall be a minimum of 50% in cohesion of the material as measured on the total area of the break. The faces of the concrete blocks to which the thermoplastic traffic line is applied shall be first sandblasted to remove laitance and primed with the Primer for Traffic Paints and Thermoplastic Traffic Paints, State Specification 63-F-40.

Yellowness Index

When tested according to Federal Test Method Standard No. 141, Method 6131, the yellowness shall not exceed 0.12 after the sample is cooled to 77° F.

Indentation Resistance

Hardness shall be measured by a Shore Durometer, Type A2, as described in ASTM D 1706-59T, except that the Durometer shall be at 75-77° F, and a 2 kilogram load applied. After 15 seconds, the reading shall be the amount herein designated when the material is cooled to the following temperatures:

<u>Temperature</u>	<u>Reading</u>
115° F	45-60

Flowability

When 400 grams of the thermoplastic traffic line is poured at a 45° angle from a cylindrical pint can approximately 3-3/4 inches high and 3-5/16 inches in diameter and with the rim removed, the residue in the can after flow ceases shall not exceed 15 percent of the original weight when the melted material is stirred rapidly for 10 seconds before pouring. The apparatus used for holding the pouring can shall be shielded with asbestos.

Low Temperature Stress Resistance (Cracking)

The thermoplastic material shall not crack or fail to adhere to the substrate when tested as follows:

A 1/8 inch thick layer of the thermoplastic material shall be applied to a one-foot square surface of a Portland cement concrete block 4 inches

thick which surface has been first sandblasted to remove all laitance and foreign matter and primed with the Primer for Traffic Paints and Thermoplastic Traffic Paints, State Specification 63-F-40. The concrete block to which the thermoplastic material has been applied shall be placed in a freezer chest or other insulated cold compartment and maintained at 15° F for 24 hours. The thermoplastic material shall be examined immediately after the concrete block is removed from the cold compartment.

After heating for four hours at 400° F the thermoplastic traffic line material shall conform to the following requirements:

Flowability

A maximum of not more than 20% residue shall remain in the can when tested according to the procedure outlined above for Flowability at 450° F.

Yellow Type

Yellow thermoplastic traffic line shall contain pigmentation giving the Color No. 1315 of Federal Specification TT-C-595 after it has been heated for four hours at 400° F and cooled to 77° F.

The physical characteristics and composition of yellow thermoplastic traffic line material shall comply in all respects with those of the white thermoplastic type except that the requirements for reflectance, resistance to yellowing and titanium dioxide content shall not apply.

Specific Gravity of Thermoplastic

The specific gravity of the white and yellow thermoplastic traffic line materials as received shall not exceed 2.15 when tested according to ASTM D 153-54, Method A. The immersion liquid shall be kerosene and the test

sample shall be ground to pass a No. 30 (U. S. Standard Sieve Series) sieve to perform the specific gravity test.

Packaging

The thermoplastic traffic line shall be packaged in suitable containers to which it will not adhere during shipment or storage. The blocks of cast thermoplastic material shall be approximately 12 x 35-1/2 x 2 inches and shall weigh approximately 50 pounds. Each container label shall designate the color, manufacturer's name, batch number and date of manufacture. Each batch manufactured shall have its own separate number.

Patents

The Contractor shall assume all costs arising from the use of patented materials, equipment, devices, or processes used on or incorporated in the work, and agrees to indemnify and save harmless the State of California, and their duly authorized representative from all suits at law or action of every nature for, or on account of the use of any patented materials, equipment, devices or processes.

APPENDIX C

Cost Calculations

Estimated Placement Cost of Hot Thermoplastics:

Equipment Basis:

Hot melt applicator cost	\$25,000
Service life	10 yrs
Time in use	800 hrs/yr
Operating speed	2 mi/hr

Computation:

$$\frac{\$25,000 \text{ appl. cost}}{10 \text{ yr serv. life}} \times \frac{1 \text{ yr opp.}}{800 \text{ hrs}} \times \frac{\text{hr}}{2 \text{ mi}} \\ = \text{Equipment Cost/mile} \quad \$1.60$$

Labor Basis:

No. of Men	3
Wages & Overhead	\$4.00/hr

Computation:

$$\frac{\$4.00}{\text{man-hr}} \times 3 \text{ men} \times \frac{\text{hr}}{2 \text{ mi}} \\ = \text{Labor Cost/mile} \quad \$6.00$$

Supplies & Maintenance Basis:

L. P. Gas/mi	\$.40
Gasoline/mi	.04
Repairs/mi	<u>1.06</u>
	\$1.50

Computation:

Suppl. & Maint. Cost/mile	<u>\$1.50</u>
TOTAL PLACEMENT COST/MILE	\$9.10

Estimated Placement Cost of Paint:

$$\frac{\$25,000 \text{ Equipment Cost}}{10 \text{ yr Service Life}} \times \frac{1 \text{ yr Period}}{800 \text{ hrs Operation}} \times \frac{\text{hr}}{10 \text{ mi}}$$

= Equipment Cost/mile \$.31

$$\frac{\$4.00}{\text{man-hr}} \times 3 \text{ men} \times \frac{\text{hr}}{10 \text{ mi}}$$

= Labor Cost/mile 1.20

Gasoline, solvents, repairs, etc.

= Suppl. & Maint. Cost/mile .30

TOTAL PAINT PLACEMENT COST/MILE \$1.81

Hot Melt Volume/mile Calculation:

$$\frac{0.1 \text{ in. (thickness)} \times 4 \text{ in. (width)} \times 5280 \text{ ft/mi} \times 12 \text{ in/ft}}{232 \text{ in}^3/\text{gal}} = 110 \text{ gal/mi}$$

Priming Cost Calculation:

<u>Materials</u>	<u>cost/lb</u>	<u>lbs</u>	<u>Density, lb/gal</u>	<u>Volume, gal</u>	<u>Cost</u>
Epon Resin 828	0.52	33.3	9.0	3.70	17.30
Epon Curing Agent V-25	1.73	16.7	8.0	2.09	12.20
Acetone	0.09	<u>50.0</u>	6.6	<u>7.58</u>	<u>4.50</u>
		100.0		13.37	34.00

Primer, cost/lb \$0.34

Density, lb/gal 7.48

$$\frac{0.005 \text{ in. (thickness)} \times 6 \text{ in. (width)} \times 5280 \text{ ft/mi} \times 12 \text{ in/ft}}{232 \text{ in}^3/\text{gal}} = 8.19 \text{ gal/mi}$$

$$8.19 \text{ gal/mi} \times 7.48 \text{ lbs/gal} \times \$0.34/\text{lb} = \$21.00/\text{mi}$$

Beading Cost Calculation:

Beading rate for paint is 6 lbs/gal of paint which yields 300 ft of stripe.

Hot melt receives the same coverage of glass beads as paint (6 lbs/300 ft of stripe)

$$\text{Glass Bead Cost/lb} = \$0.18$$

$$\frac{6 \text{ lbs} \times 5280 \text{ ft/mi} \times \$0.18/\text{lb}}{300 \text{ ft}} = \$19.00/\text{mi}$$

Paint Cost Calculation:

Paint Cost/gal 1.53

$$\frac{.015 \text{ in. (thickness)} \times 4 \text{ in. (width)} \times 5280 \text{ ft/mi} \times 12 \text{ in/ft}}{232 \text{ in}^3/\text{gal}} = 16.39 \text{ gal/mi}$$

$$16.39 \text{ gal/mi} \times \$1.53/\text{gal} = \$25.00/\text{mi}$$

TABLE XXV
SUMMARY OF MATERIALS COSTS

Materials	cost/lb	26		27		28		155		157		158	
		lbs	cost	lbs	cost	lbs	cost	lbs	cost	lbs	cost	lbs	cost
Abitol	0.36	348	125.30	340	122.40								
Stabilite 10	0.23	588	135.20	590	135.70	590	135.70	738	169.70	443	101.90	1107	254.60
Pliolite ACL, milled	0.49	540	264.60										
VBR 5010	0.35					590	206.50						
Pentlyn K	0.31					296	91.80						
Ethocel N-4	0.86			546	469.60								
Pentylphenol	0.16			6	1.00								
Polymid 1560	1.08							553	597.20	775	837.00	280	302.40
Santizer 8	0.52							185	96.20	258	134.20	89	46.30
Glass Beads	0.18	1250	225.00	1250	225.00	1250	225.00	1510	271.80	1510	271.80	1510	271.80
Titanium Dioxide R-610	0.27	780	210.60	780	210.60	780	210.60	210	56.10	210	56.70	210	56.70
Calcium Carbonate R0-40	0.02	2500	50.00	2500	50.00	2500	50.00	1300	26.00	1300	26.00	1300	26.00
Calcium Carbonate X0	0.02							1510	30.20	1510	30.20	1510	30.20
Total		6006	1010.70	6012	1214.30	6006	919.60	6006	1247.80	6006	1457.80	6006	988.00
Materials Cost/lb			0.168		0.202		0.153		0.208		0.243		0.164
Manufacturing Cost/lb			<u>0.03</u>		<u>0.03</u>		<u>0.03</u>		<u>0.03</u>		<u>0.03</u>		<u>0.03</u>
Hot Melt Cost/lb			0.198		0.232		0.183		0.238		0.273		0.194
Density, lb/gal			16.4		16.5		16.9		16.3		16.6		16.3
lbs/mile stripe ¹ (@ 110 gal/mile)			1804		1815		1859		1793		1826		1793
Hot Melt Cost/mile			357.00		421.00		340.00		427.00		498.00		348.00
Priming Cost/mile ²			21.00		21.00		21.00		21.00		21.00		21.00
Beading Cost/mile ³			19.00		19.00		19.00		19.00		19.00		19.00
All Materials Cost/mile			397.00		461.00		380.00		467.00		538.00		388.00

¹See Volume/mile Calculation, page 182.

²See Priming Cost Calculation, page 182.

³See Beading Cost Calculation, page 182.

TABLE XXV (Concluded)
SUMMARY OF MATERIALS COSTS

Materials	cost/lb	160 lbs cost	162 lbs cost	163 lbs cost	Max.	Economy	Proprietary A cost	Ga. Spec. Paint cost
Abitol	0.36	348	125.30					
Stabilite 1C	0.23	588	135.20	536	123.30	531	122.10	
Fluolite ACL, milled	0.49	540	264.60					
VBR 5010	0.35			536	187.60	620	217.00	
Pentlyn K	0.31			403	124.90	324	100.40	
Ethocel M-4	0.86							
Pentylphenol	0.16							
Polyimid 1560	1.08							
Santizer 8	0.52							
Glass Beads	0.18	1250	225.00	1510	271.80	1510	271.80	
Titanium Dioxide R-610	0.27	780	210.60	210	56.70	210	56.70	
Calcium Carbonate RO-40	0.02	2500	50.00	1300	26.00	1300	26.00	
Calcium Carbonate XO	0.02			1510	30.20	1510	30.20	
Total		6006	1010.70	6005	820.50	6005	824.20	
Materials Cost/lb			0.168		0.137		0.137	
Manufacturing Cost/lb			0.03		0.03		0.03	
Hot Melt Cost/lb			0.198		0.167		0.167	0.50
Density, lb/gal			16.4		17.0		17.0	18.1
lbs/mile stripe (@ 110 gal/mile)			1804		1870		1870	1991
Hot Melt Cost/mile			357.00		312.00		312.00	234.00 ⁴
Priming Cost/mile			21.00		21.00		21.00	21.00
Bonding Cost/mile			<u>19.00</u>		<u>19.00</u>		<u>19.00</u>	<u>19.00</u>
All materials Cost/mile			397.00		352.00		352.00	274.00

⁴See Paint Cost Calculation, page 182.

APPENDIX D

References Cited

REFERENCES CITED

- (1) Herman F. Mark, "New Concepts--New Polymers--New Applications," American Scientist 55, No. 3, 265-281 (September 1967).
- (2) W. R. Tooke, Jr. and W. H. Burrows, Laboratory Wear Testing of Traffic Paints, Final Technical Report, Part III, 39-63, Project B-210, Engineering Experiment Station of the Georgia Institute of Technology, Atlanta, Georgia, September 30, 1965, 104 pages.
- (3) J. M. Dalla Valle, Micromeritics, Chapter 6, Pitman Publishing Corp., New York (1948).

NOTE: References pertaining to a literature review are cited in the context of Section III, State of the Art Survey.